

STUDY OF THE CHARACTERISTICS OF THE CORROSION  
FILM ON ZIRCONIUM USING POLARIZED LIGHT

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May 16, 1952

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Massachusetts Institute of Technology  
Cambridge, Massachusetts

Dear Sir:

In accordance with the requirements for the Degree of  
Master of Science, we submit herewith a thesis entitled, "Study of  
the Characteristics of the Corrosion Film on Zirconium using Polarized  
Light."

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STUDY OF THE CHARACTERISTICS OF THE CORROSION FILM ON ZIRCONIUM  
USING POLARIZED LIGHT

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ACKNOWLEDGMENT

The authors wish to express their appreciation to Professor A. R. Kaufmann for his suggestion of the method used in this study and for his guiding advice during the progress of this work. In addition, the wholehearted cooperation and enthusiastic support of the entire M.I.T. Metallurgical Project contributed materially to the completion of this project in the relatively short time available.

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ABSTRACT  
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STUDY OF THE CHARACTERISTICS OF THE CORROSION FILM ON  
ZIRCONIUM USING POLARIZED LIGHT

by

Commander Nels Roland Nelson, U. S. Navy

and

Lieutenant John Wade Heintz, U. S. Navy

Submitted for the degree of Master of Science in the  
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While pure zirconium is virtually non-corrosive at low temperatures, its corrosion-resistance at high temperatures varies over a wide range depending upon the amount and type of impurities present. The purpose of this thesis was to study with polarized light the effect of nitrogen on the corrosion-resistance of zirconium in distilled water at 450°F and at 560°F.

Six specimens of zirconium (essentially the same in chemical analysis except for variations in nitrogen content) were studied with plane polarized light. The data obtained consisted of measurements of the phase shift and rotation of the plane of polarization of the light reflected from the surface of the specimens during a series of incremental corrosion film growths.

The characteristic angle of zirconium in the electropolished condition was observed to be  $295^{\circ} - 03' \pm 1^{\circ} - 39'$ . In general, with successive

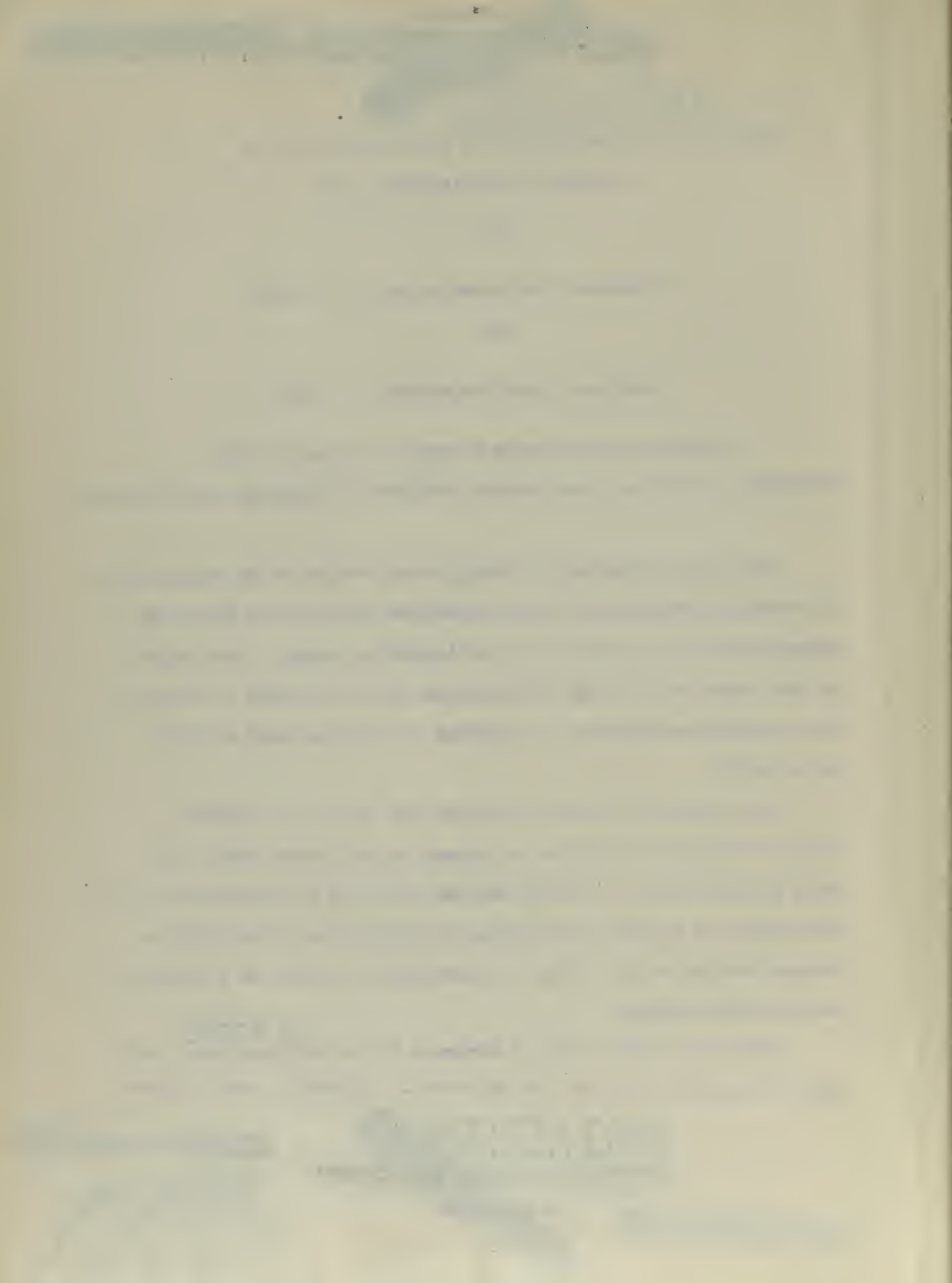
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additions of corrosion film, the characteristic angle decreased for each specimen. An exception is made to this statement for those specimens which had their optic axes normal to the surface.

Previous work in this field has shown that an increase in nitrogen causes a decrease in the corrosion-resistance of zirconium. The results of the studies made in this thesis do not indicate that either the characteristic angle,  $\theta$ , (or any other observable parameter) vary in a manner consistent with nitrogen content. Consequently, without additional investigation, polarized light cannot be used to test the corrosion-resistance of a random zirconium sample.

With refined methods of reducing the amount of miscellaneous impurities in the zirconium specimens to be studied, with reduction of errors in the optical equipment, and with the incorporation of a preferred crystal orientation, it is probable that polarized light may still be used to determine the corrosion-resistance of zirconium.

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TABLE OF CONTENTS

	<u>Page</u>
Abstract	i
List of Illustrations	iv
List of Tables	vi
Chapter I	Introduction
	1
Chapter II	Theoretical Analysis
	3
Chapter III	Equipment and Procedure
	22
Chapter IV	Experimental Results and Conclusions
	38
Chapter V	Recommendations for Further Investigation
	44
Appendix A	Geometrical Analysis of Poincare Sphere for Phase Shift due to Specimen
	46
Appendix B	Calculation of Nitrogen Addition to Specimens
	48
Appendix C	Chemical and Spectrographical Analysis
	50
Appendix D	Data
	52
Appendix E	Summary of Data
	199
Appendix F	Calculation of Characteristic Angle
	202
Bibliography	208

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- iii -

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LIST OF ILLUSTRATIONS

		<u>Page</u>
Figure II-1	Resolution of Plane Polarized Light into Components Along Principal Directions of Crystal	4
Figure II-2(a)	Plane Polarized Light	4
Figure II-2(b)	Elliptically Polarized Light	4
Figure II-3(a)	Components of Electric Vector of Incident Plane Polarized Light Along Principal Direction. Plane of Polarization at Angle $\alpha$ with Principal Direction	7
Figure II-3(b)	Components of Electric Vector of Incident Plane Polarized Light Along Principal Direction. Plane of Polarization Coincident with Principal Direction	7
Figure II-4(a)	Components of Electric Vector of Incident Plane Polarized Light Along Principal Directions that are Observed Through Crossed Analyzer. Plane of Polarization at Angle $\alpha$ with Principal Directions.	7
Figure II-4(b)	Components of Electric Vector of Incident Plane Polarized Light Along Principal Directions that are Observed Through Crossed Analyzer. Plane of Polarization Coincident with Principal Directions.	7
Figure II-5	Path of Elliptically Polarized Light Inscribed in Rectangle with Ellipticity of $\rho = \tan \psi$	7
Figure II-6	Path of Elliptically Polarized Light. A leads B by angle $\phi$ . $\phi$ is Azimuth of Elliptical Light.	13
Figure II-7	Poincare Sphere	13
Figure II-8	Schematic Diagram of Bausch and Lomb Elliptical Compensator	17
Figure II-9	Phase Shift and Rotation Variations Introduced in Plane Polarized Light by Elliptical Compensator and Specimen Illustrated on Poincare Sphere	17
Figure III-1	Vacuum Drip Melting Apparatus	25

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- iv -





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Figure III-2	M.I.T. Copper Crucible	24
Figure III-3	Zirconium Billet After Having Been Drip Melted into Copper Crucible	24
Figure III-4	Sectioning of Slice G-G to Obtain Specimen H	24
Figure III-5	Alignment and Photomultiplier Arrangement	29
Figure III-6	Metallograph Optics	31
Figure III-7	Metallograph and Associated Equipment with Elliptical Compensator in Place	32
Figure III-8	Rausch and Lomb Elliptical Compensator	33
Figure IV-1	Summary Plot of Characteristic Angle $\tau$ for Run No. 1	39
Figure IV-2	Summary Plot of Characteristic Angle $\tau$ for Run No. 2	40
Figure A-1	Projection View of Poincare Sphere Showing Phase Shift Introduced in Inci- dent Light by Specimen	47
Figure B-1	Typical Laboratory Apparatus for Adding Nitrogen to Zirconium	49

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Table C-1

Chemical Content of Parent Zirconium and of  
Six Samples Made Therefrom

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Chapter I

INTRODUCTION

Because zirconium has a low cross-section for thermal neutrons and has desirable structural qualities, it is being used more and more in nuclear reactors. In this application, the zirconium often is exposed to relatively high temperature distilled water for long periods of time. Consequently, its corrosion-resistance under these conditions has become important, especially since it varies over a wide range depending upon the amount and type of impurities present. To date, no reliable test has been developed which will give a rapid, accurate evaluation of the corrosion-resisting quality of a random sample of zirconium.

The purpose of this thesis was to study with polarized light the effect of nitrogen on the corrosion-resistance of Westinghouse Grade I Crystal Bar Zirconium. The particular type of corrosion investigated was that caused by surface contact with distilled water at 450° F and at 560° F. The study was limited to areas of single grains which did not include any grain boundaries.

Modern research metallographs provide an excellent means of using polarized light to examine the grain structure of hexagonal metals such as zirconium. The optical effects involved in such an examination of the reflection of polarized light from opaque surfaces have been known for over a half-century. More recently, the development of an Elliptical Vibration Compensator has made possible an easy method of accurately

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measuring these effects. A detailed discussion of the theory involved in this method is contained in Chapter II.

While a survey of the literature has revealed no previous attempts to use polarized light to study zirconium corrosion films, a small amount of information is available concerning related problems. Most of this information is summarized by B. W. Mott and H. N. Haines<sup>7</sup> in "The Examination of Metals Under Polarized Light," Part I, AERE, M/R 604. The latest previous work of this nature is a Bachelor of Science thesis entitled "High Sensitivity Measurements of the Optical Anisotropy of Beryllium" by Edward L. Bronson at M.I.T. in 1951. He found appreciable changes in elliptical compensator readings for different types of films on beryllium. This result suggested to Dr. A. R. Kaufmann that the characteristics of the corrosion film of zirconium might be studied in a similar manner and that possibly the results might provide a means for devising a corrosion-resistance test. Preliminary work by the authors during the M.I.T. fall semester supported this belief and precipitated the present study.

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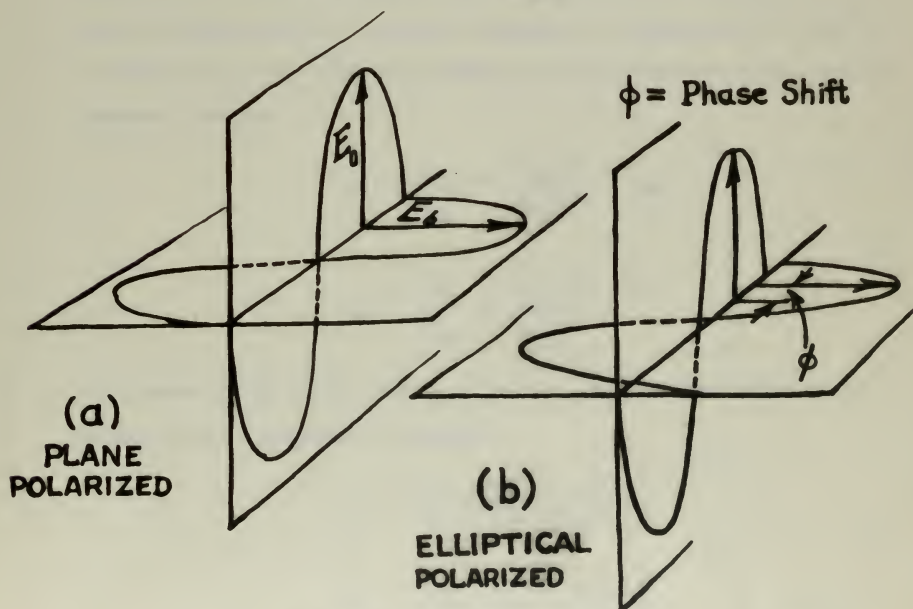
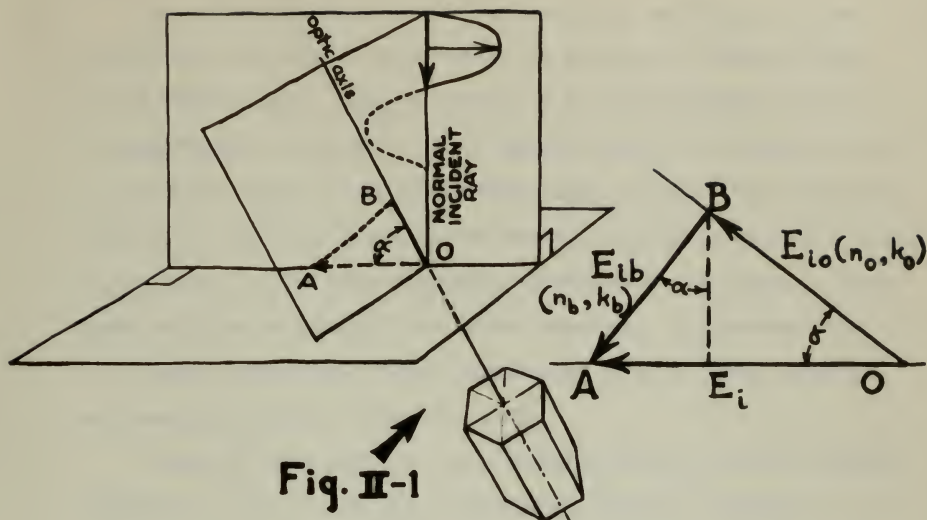
Chapter IITHEORETICAL ANALYSIS

At room temperature zirconium is a close packed hexagonal crystal. The optical properties of such crystals are known to vary for different directions in the crystal and are therefore said to be optically anisotropic. In the hexagonal system there are two crystallographic planes of symmetry -- one containing the major crystallographic axis or optic axis,  $c$ ; and the plane that is normal to this containing the basal plane. These two directions are called the principal directions of the crystal. The optical properties of the crystal in each of these directions can be described by its refractive index,  $n$ , and absorption coefficient,  $k$ . Thus  $n_o$  and  $k_o$  describe the optical characteristics of the crystal in the direction of the optic axis and  $n_p$  and  $k_p$  describe the characteristics in a direction perpendicular to the optic axis.

When a ray of plane polarized light falls upon the polished surface of a zirconium specimen, the vibrating vector of this light can be resolved into two components -- one lying in the plane containing the optic axis and the other perpendicular to this. Since all experimental work carried out in this thesis was done with normal vertical illumination on zirconium specimens, all figures and descriptions will be based on this. Figure II-1 shows the ray of plane polarized light at normal incidence to a zirconium specimen whose optic axis is neither in the same plane as the normal to the surface nor in the plane containing the vibrating electric vector of the incident light.  $E_i$  is the incident electric vector with component  $E_{i0}$  along the optic axis and  $E_{ip}$  perpendicular to the optic axis.

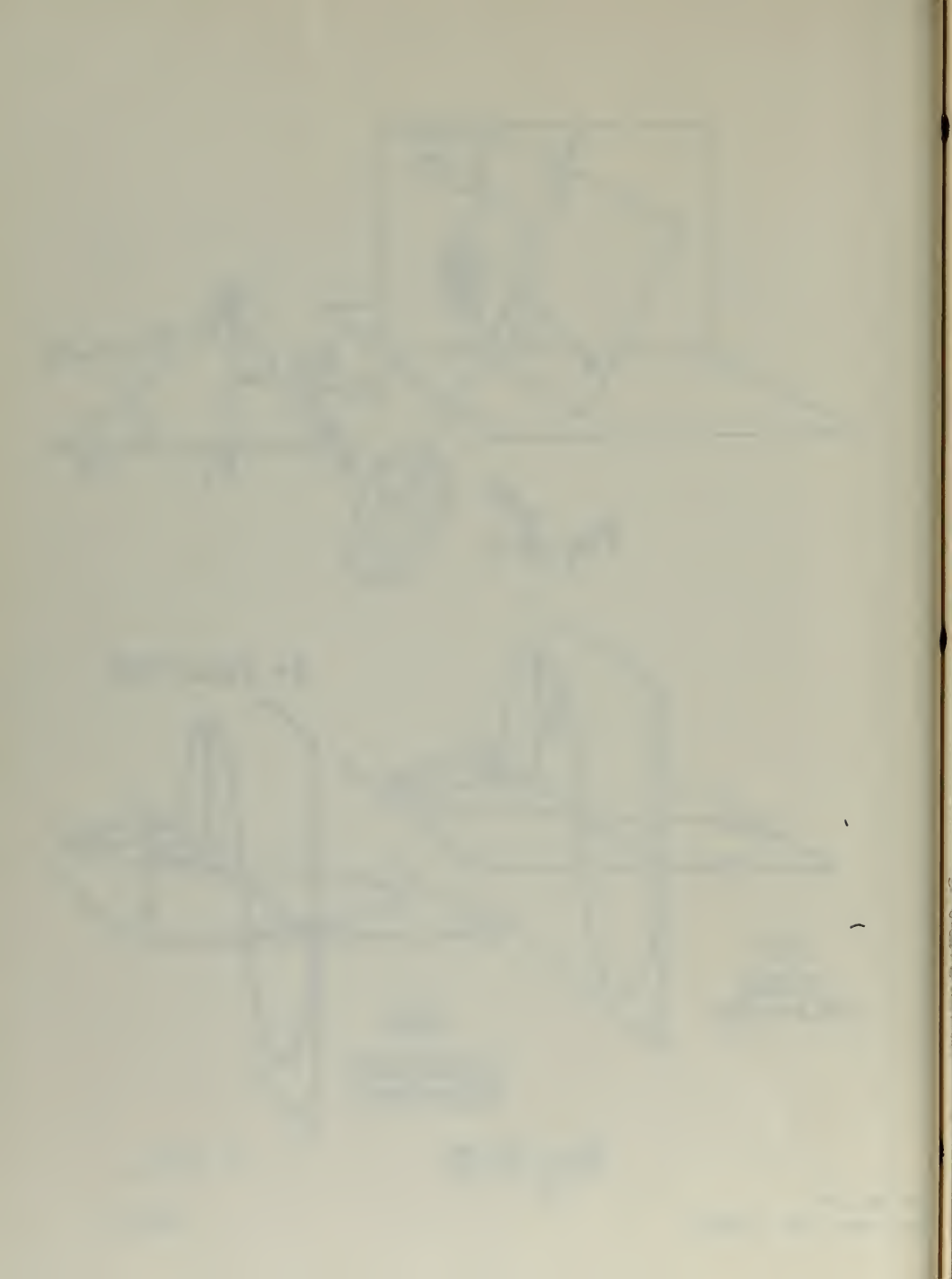
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**Fig. II-2**

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The refractive index,  $n$ , and the absorption coefficient,  $k$ , are properties of the crystal which affect the intensity or energy carried by the incident wave. Since the energy of a plane polarized light wave is proportional to the square of the electric vector, the electric vector,  $E_1$ , and its components will be correspondingly affected. The properties,  $n$  and  $k$ , in a specific direction are referred to as an index pair -- being  $n_o, k_o$  and  $n_b, k_b$  for their respective directions in the crystal. These index pairs may be thought of as vector operators. In zirconium which is an opaque (absorbing) crystal, these index pairs or vector operators are complex operators of the form  $(n-jk)$ .

Since the index pair,  $n_o, k_o$  is different than  $n_b, k_b$ , the incident component  $E_{1b}$  will be operated on differently than the component  $E_{1o}$ . If the reflecting power of a principal direction is defined as the ratio of the intensity of the reflected component to the intensity of the incident component, we have

$$\left. \begin{aligned} R_o &= \frac{I_{ro}}{I_{1o}} = \frac{(E_{ro})^2}{(E_{1o})^2} \quad \text{and} \\ R_b &= \frac{I_{rb}}{I_{1b}} = \frac{(E_{rb})^2}{(E_{1b})^2} \end{aligned} \right\} \text{----- (1)}$$

or in terms of the index pairs  $n_o, k_o$  and  $n_b, k_b$  when the medium of immersion is air, equation (1) becomes<sup>4</sup>

$$\left. \begin{aligned} R_o &= \frac{(n_o - 1)^2 + n_o^2 k_o^2}{(n_o + 1)^2 + n_o^2 k_o^2} \\ R_b &= \frac{(n_b - 1)^2 + n_b^2 k_b^2}{(n_b + 1)^2 + n_b^2 k_b^2} \end{aligned} \right\} \text{----- (2)}$$





Thus when the incident vector component,  $E_{i0}$ , is operated on by  $R_0$  during reflection, the reflected component,  $E_{r0}$ , will be different. Similarly when  $E_{ib}$  is operated on by  $R_b$ ,  $E_{rb}$  will be different. Both components of the incident vector will have been changed in magnitude and will be out of phase with each other when reflected. That is, while in the specimen the change in velocity of propagation of  $E_{i0}$  due to  $R_0$  was not the same as the change introduced in  $E_{ib}$  by  $R_b$  and upon emergence the reflected components were out of phase with each other.

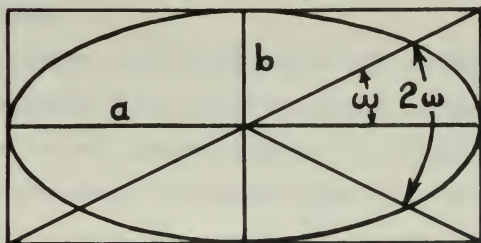
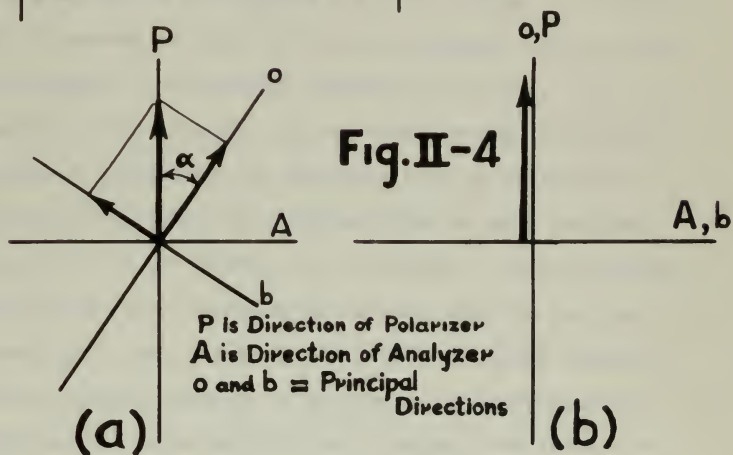
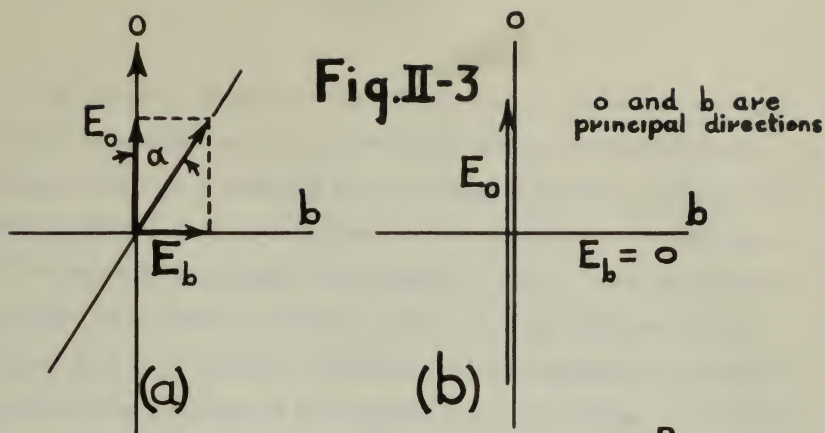
In Figure II-2(a) the incident ray is shown resolved into two components at right angles to each other and in Figure II-2(b) the reflected ray is shown resolved into two components at right angles to each other. Thus the incident plane polarized light is reflected as elliptically polarized light due to the optical anisotropy of zirconium.

In the special case where the electric vector,  $E_i$ , is coincident with one of the principal directions such as the optic axis, there will be no component in the direction of the basal plane, i.e.,  $E_{ib}$  will be zero.  $E_{i0}$  will equal  $E_i$  and the resulting reflected light will be linearly polarized. Summarizing, when plane polarized light is incident (normal) on a specimen of zirconium, elliptically polarized light will be reflected except when the direction of polarization is coincident with a principal direction. This can be better visualized in Figure II-3. In Figure II-3(a), component  $E_0$  is operated on by  $R_0$  and  $E_b$  by  $R_b$ . This changes  $E_0$  and  $E_b$  so that they form elliptically polarized light. For each angle  $\alpha$ , the resulting elliptical light will be different and the maximum ellipticity will occur for  $\alpha = 45^\circ$ . In Figure II-3(b) component  $E_0$  is operated on by  $R_0$  and since  $E_b = 0$ , the reflected light will be linearly polarized in the same direction.

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**Fig. II-5**

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Now consider the case in which the specimen is viewed through an analyzer whose direction is at right angles to the polarizer supplying the light. This is a condition known as "crossed nicols." Figure II-4(a) shows the direction of the polarizer, P, the direction of the analyzer, A, and the principal directions of the crystal, o and b. When the specimen is rotated to an angle  $\alpha$  between P and o, the light reflected to the analyzer will be elliptically polarized and the components of this light parallel to the direction of the analyzer will pass through. As  $\alpha$  varies when the stage is rotated the ellipticity of the reflected light and thus the components parallel to the analyzer direction will change. This will vary the intensity of the reflected light as viewed through the analyzer. When the direction of polarization is coincident with the principal direction, o, as in Figure II-4(b) the reflected light is plane polarized and is at right angles to the direction of the analyzer. No light should be transmitted in this case. Therefore as the stage with the specimen is rotated through  $560^\circ$  there should be four points of minimum intensity and four points of maximum intensity -- the minimum occurring when the principal directions are coincident with the polarizer direction and the maximum when the ellipticity is the greatest, i.e., when the angle  $\alpha$  is  $45^\circ$  (diagonal position).

It is noted, however, during observations that the minimum intensities are never complete nulls as would be expected and that the four maximum intensities are not of equal magnitude. As brought out by Berek<sup>4</sup>, with physical optical systems it is almost impossible to provide a source of pure plane polarized light to the specimen. The optical systems

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usually create a small amount of ellipticity and therefore even when the principal directions are coincident with the direction of polarization as in Figure II-4(b), some light can be viewed through the analyzer.

If devices, which can compensate for the ellipticity introduced by zirconium into plane polarized light, can be inserted in the path of the elliptical light, it is believed that much information can be obtained about the surface properties of zirconium. This ellipticity is caused by a phase shift and the rotation of the plane of polarization. Therefore the ellipticity could be removed by two compensating devices of known characteristics -- one which removes the phase shift and the other which removes the rotation. To see how this can be done, the geometry of elliptically polarized light should be reviewed.\*\*

The locus of a point tracing an elliptical path is given by

$$\left. \begin{aligned} y &= R \sin \frac{2\pi t}{T} \\ x &= A \sin \frac{2\pi t}{T} + \phi \end{aligned} \right\} \text{----- (3)}$$

where the x component leads the y component by an angle  $\phi$  (in radians). R is the amplitude of the y component (retarded) and A is the amplitude of the x component (accelerated). T is the period of vibration and t is the time elapsed since  $y = 0$ .

Referring to Figure II-5, the ellipticity is defined as

$$\int = \frac{b}{a} = \tan \omega \text{----- (4)}$$

or 
$$\sin 2\omega = \frac{2\int}{1 + \int^2} \text{----- (5)}$$

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\*\* The figures and nomenclature used are those of C. A. Skinner<sup>6</sup>.

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funds to carry out its policy.

and the equation of an ellipse referred to its own axes is

$$\frac{x^2}{a^2} + \frac{y^2}{b^2} = 1 \quad \text{----- (6)}$$

In Figure II-6, consider an elliptical pattern of light where  $x$  is the incident vibration direction.  $\theta$  is defined as the azimuth of the elliptical light.

$$\frac{x'^2}{a^2} + \frac{y'^2}{b^2} = 1 \quad \text{----- (7)}$$

and

$$A = x_1' \cos \theta - y_1' \sin \theta \quad \text{----- (8)}$$

Combining (7) and (8)

$$A^2 = a^2 \cos^2 \theta + b^2 \sin^2 \theta \quad \text{----- (9)}$$

$$R^2 = a^2 \sin^2 \theta + b^2 \cos^2 \theta \quad \text{----- (10)}$$

adding and subtracting (9) and (10)

$$A^2 + R^2 = a^2 + b^2 \quad \text{----- (11)}$$

$$\begin{aligned} A^2 - R^2 &= (a^2 - b^2) (\cos^2 \theta - \sin^2 \theta) \\ &= (a^2 - b^2) \cos 2\theta \quad \text{--- (12)} \end{aligned}$$

By introducing the phase lead,  $\phi$ , of the  $A$  component over the  $R$  component, other fundamental relations can be derived. From equation (3), if  $y = 0$  and  $x = p$ , we have

$$p = A \sin \phi \quad \text{----- (13)}$$

From Figure II-6, this point  $p$  is  $x_2'$ ,  $y_2'$  and

$$x_2' = p \cos \theta = A \sin \phi \cos \theta \quad (14)$$

$$y_2' = p \sin \theta = A \sin \phi \sin \theta \quad (15)$$

D 52003

Jan 2. 1874. Sunday. Clear. Cold.

Jan 3. 1874. Monday. Clear. Cold.

Jan 4. 1874. Tuesday. Clear. Cold.

Jan 5. 1874. Wednesday. Clear. Cold.

Jan 6. 1874

Jan 7. 1874. Thursday. Clear. Cold.

Jan 8. 1874. Friday. Clear. Cold.

Jan 9. 1874. Saturday. Clear. Cold.

Jan 10. 1874. Sunday. Clear. Cold.

Jan 11. 1874. Monday. Clear. Cold.

Jan 12. 1874. Tuesday. Clear. Cold.

Jan 13. 1874. Wednesday. Clear. Cold.

Jan 14. 1874. Thursday. Clear. Cold.

Jan 15. 1874. Friday. Clear. Cold.

Jan 16. 1874. Saturday. Clear. Cold.

Jan 17. 1874. Sunday. Clear. Cold.

Jan 18. 1874. Monday. Clear. Cold.

Jan 19. 1874. Tuesday. Clear. Cold.

Jan 20. 1874. Wednesday. Clear. Cold.

Jan 21. 1874. Thursday. Clear. Cold.

Jan 22. 1874. Friday. Clear. Cold.



Substituting (14) and (15) into (6) gives

$$A^2 \sin^2 \phi = \frac{a^2 b^2}{a^2 \sin^2 \theta + b^2 \cos^2 \theta} \quad \text{-- (16)}$$

and from (10)  $AR \sin \phi = ab \quad \text{-- (17)}$

We have further

$$\cos^2 \theta = 1 - \sin^2 \theta = 1 - \frac{a^2 b^2}{(a^2 \cos^2 \theta + b^2 \sin^2 \theta)(a^2 \sin^2 \theta + b^2 \cos^2 \theta)}$$

or  $\cos^2 \phi = \frac{(a^2 - b^2)^2 \sin^2 \theta \cos^2 \theta}{A^2 R^2}$

or  $AR \cos \phi = \frac{a^2 - b^2}{2} \sin 2\theta \quad \text{-- (18)}$

Rewriting equations (11), (12), (17), and (18) and defining

$$P \equiv \frac{A^2 + R^2}{2} \equiv \frac{a^2 + b^2}{2} \quad \text{-- (19)}$$

$$Q \equiv \frac{A^2 - R^2}{2} = \frac{a^2 - b^2}{2} \cos 2\theta \quad \text{-- (20)}$$

$$K \equiv AR \cos \phi = \frac{a^2 - b^2}{2} \sin 2\theta \quad \text{-- (21)}$$

$$S \equiv AR \sin \phi = ab \quad \text{-- (22)}$$

Combining (19), (20), (21), (22) gives

$$Q^2 + K^2 + S^2 = P^2 \quad \text{-- (23)}$$

which suggests the equation for a sphere in which Q, K, and S are the rectangular coordinates whose radius is P with the center at the origin.

NOTES ON THE  
ANTHROPOLOGY OF THE  
INDIAN RACES

BY  
J. H. HENNESSY, M.A., F.R.S.,  
FELLOW OF THE ROYAL SOCIETY

IN THE  
MUSEUM OF THE  
HUMAN SPECIES

THE  
JOURNAL OF THE  
ROYAL ANTHROPOLOGICAL INSTITUTE

FOR THE  
YEAR 1885

THE  
JOURNAL OF THE  
ROYAL ANTHROPOLOGICAL INSTITUTE

FOR THE  
YEAR 1886

THE  
JOURNAL OF THE  
ROYAL ANTHROPOLOGICAL INSTITUTE

FOR THE  
YEAR 1887

THE  
JOURNAL OF THE  
ROYAL ANTHROPOLOGICAL INSTITUTE

FOR THE  
YEAR 1888

To show how the sphere may be used to determine the constants of an elliptic vibration we have for the azimuth  $\Theta$  of the ellipse from (20) and (21)

$$\tan 2\Theta = \frac{K}{Q} \quad \text{-----} \quad (24)$$

in which  $\Theta$  can assume any value between 0 and  $\pi$  and is independent of  $S$ . Similarly from (19) and (22)

$$\frac{2ab}{a^2 + b^2} = \frac{S}{P} \quad \text{-----} \quad (25)$$

and since  $\int = \frac{b}{a}$  from (4)

$$\frac{2\int}{1 + \int^2} = \frac{S}{P} \quad \text{-----} \quad (26)$$

and from (5)  $\frac{S}{P} = \sin 2\omega$  ----- (27)

so  $\int = \tan \omega$  ----- (28)

Therefore the ellipticity,  $\int$ , is independent of  $Q$  and  $K$  and since  $\sin 2\omega$  can have any value between -1 and +1,  $\int$  may range between -1 and +1.

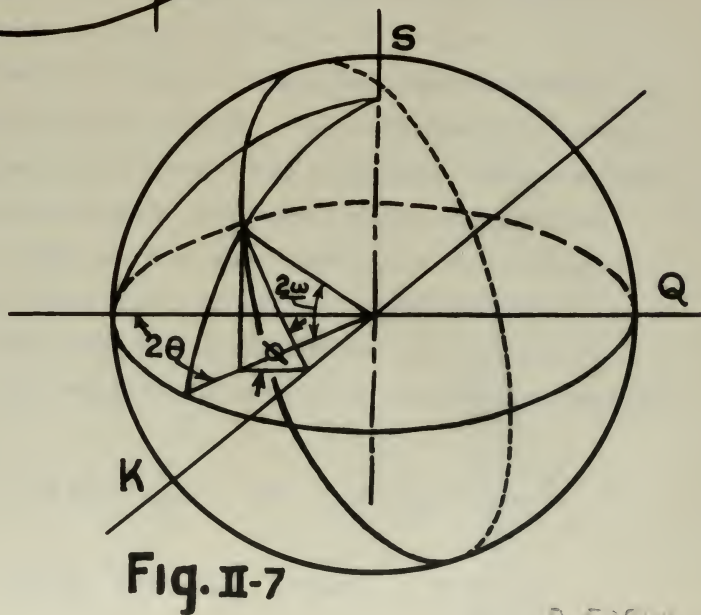
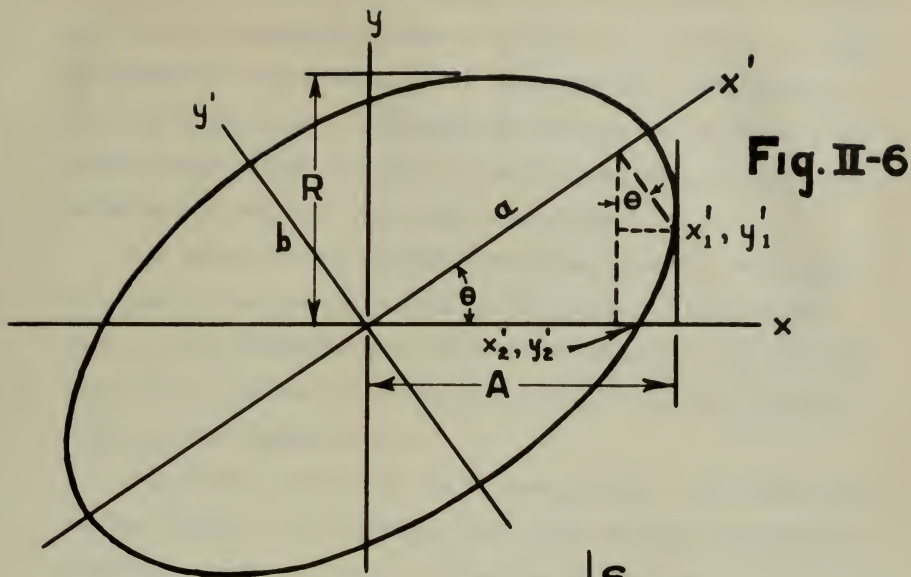
Further, the phase lead  $\phi$  of any A component, from (21) and (22) is given by

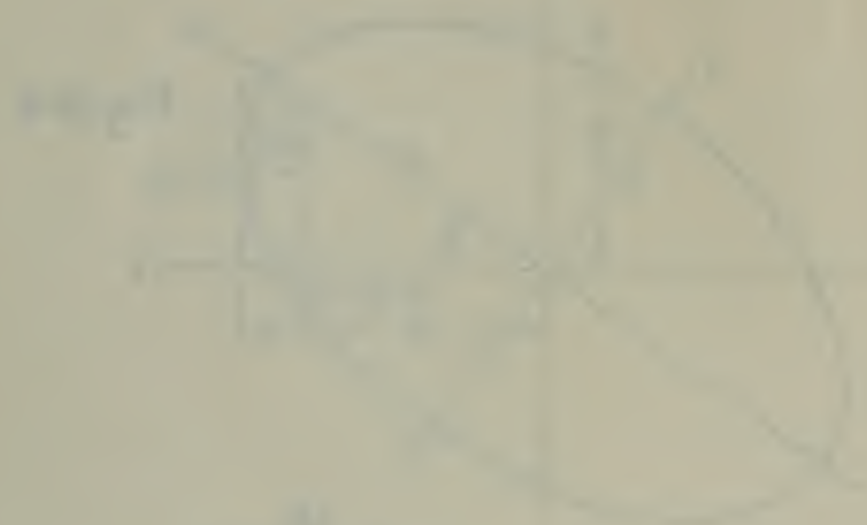
$$\tan \phi = \frac{S}{K} \quad \text{-----} \quad (29)$$

In Figure II-7, let the QK plane be the equatorial plane of the sphere and the S axis be the polar axis. Let longitude be measured from the Q axis eastward, and the latitude upward from the equatorial plane. Inspection shows that  $2\Theta$  is the longitude and  $2\omega$  is the latitude of the

D 52003







expressed vibration. Therefore the longitude on this sphere of reference can be used to determine the azimuth of the vibration while its latitude can be used to determine ellipticity or phase shift. Plane polarized light (no ellipticity) is represented by the sum total of points on the equator while positive and negative circular polarized light are represented by the north and south poles respectively.

The Bausch and Lomb Elliptical Vibration Compensator was used in this thesis to measure the rotation and ellipticity of the reflected light. In the compensator unit there are two elements: a rotation compensator and an elliptical compensator. Figure II-3 shows a schematic diagram of the compensator unit.

The rotation compensator will be taken up first. The rotation compensator consists of two identical glass prisms cemented together with a transparent cement of the same index as that of the glass. On the inclined inner face of one prism is a quarter-wave interference with an index differing from that of the glass. This quarter wave interference is tin oxide applied by high vacuum thermal evaporation. This quarter-wave is for a specific wave length and a correction must be made if other wave lengths of light are to be used. Referring to Figure II-3, it is seen that the inclined film of the rotation compensator transmits the p-component of the incident polarized light more copiously than the s-component. This results in a rotation of the polarized light traversing the film.

$$\tan \psi_d = \frac{D_p}{D_s} \quad \text{and} \quad \tan \psi_e = \frac{E_p}{E_s}$$

$$\tan \psi_d = R \tan \psi_e \quad \text{where} \quad R = \frac{D_p E_s}{D_s E_p}$$

D 52003





If the azimuth of the incident light on the film is  $\psi_e$  and the azimuth of the transmitted light is  $\psi_d$ , then with no mica plate present,  $\psi_d$  would also be the azimuth of the light incident on the specimen. If the specimen produces a rotation of the incident light, the reflected light will have an azimuth  $\psi_e$ , and after passing through the compensator film the light will be rotated again and emerge with an azimuth of  $\psi_d$ . Again

$$\tan \psi_d = R \tan \psi_e,$$

With the polarizer and analyzer crossed, compensation is obtained by turning the film about the optical axis of the system until extinction occurs. At this point compensation has been effected. Therefore at compensation with the compensator setting at  $r$ ,

$$r = \psi_e = \psi_d,$$

The rotation produced by the specimen is  $(\psi_d - \psi_e)$ .

$$\tan (\psi_d - \psi_e) = \frac{\tan \psi_d - \tan \psi_e}{1 + \tan \psi_d \tan \psi_e},$$

$$\tan (\psi_d - \psi_e) = \frac{R^2 - 1}{2R} \sin 2r$$

$$\text{or} \quad \epsilon = \frac{R^2 - 1}{2R} \sin 2r \quad \text{--- (30)}$$

where

$$\tan (\psi_d - \psi_e) = \epsilon$$

Since the rotation produced by the specimen is usually much less than  $5^\circ$

$$\epsilon \approx (\psi_d - \psi_e) = \frac{R^2 - 1}{2R} \sin 2r \quad \text{--- (31)}$$

THE UNIVERSITY OF CHICAGO

DEPARTMENT OF CHEMISTRY

RESEARCH REPORT

ON THE CHEMISTRY OF THE

HYDROLYSIS OF CERTAIN

ESTERS OF CERTAIN

## ACID

BY

JOHN EDGAR HEDGECOCK

AND

WILLIAM HENRY PERKINS

1910

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1910

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1910

In Figure II-8 the elliptical compensator is located above the rotation compensator and consists of an extremely thin mica plate cemented between cover glasses and is rotatable in its own plane. As in the rotation compensator, light traverses the mica plate in both the incident and reflected directions, thereby removing half the phase shift due to the specimen in the incident beam and the other half from the reflected beam. Since the relationship of elliptically polarized light to a sphere has already been shown, it is easy to present a geometrical explanation of what happens to light as it traverses the elliptical compensator.

Figure II-9 shows the five operations on the incident plane polarized light. The fast axis (principal direction) of the specimen is assumed to be at  $45^\circ$  to the direction of polarization. This will be a longitude of  $90^\circ$  in the sphere since all angles are double. (It should be recalled that the maximum ellipticity due to the specimen occurs when the direction of polarization of the incident light is at  $45^\circ$  to one of the principal directions of the crystal. This will fix the position of the specimen on the stage in the procedure to be described later).

Figure II-9(a) shows the rotation of the incident light after passing through the rotation compensator (inclined quarter-wave film). Figure II-9(b) shows the phase shift introduced into the rotated plane polarized light after having passed through the mica plate of the elliptical compensator. This is the light which falls on the specimen. It is to be noted that this light is now slightly elliptical having been given both a rotation and a phase shift. Figure II-9(c) shows the rotation and phase shift given to the incident light by the specimen. Figure II-9(d) shows the phase shift given to the light as it passes back through the



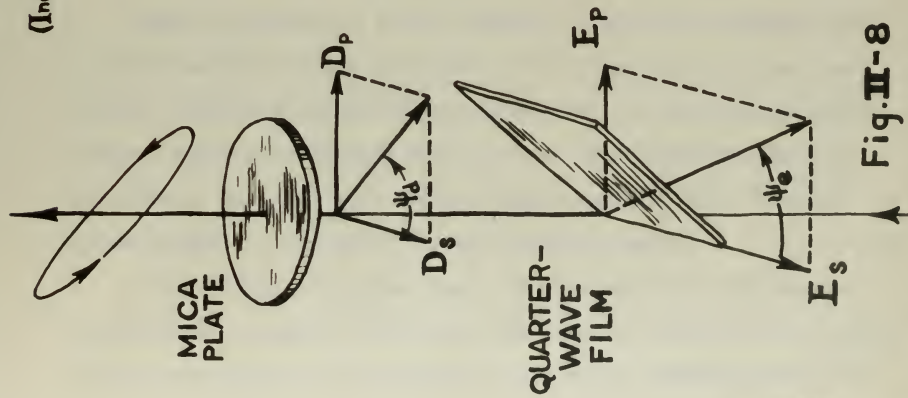
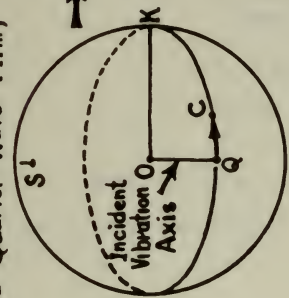


Fig. II-8

Rotation Compensator  
(Inclined Quarter-Wave Film)

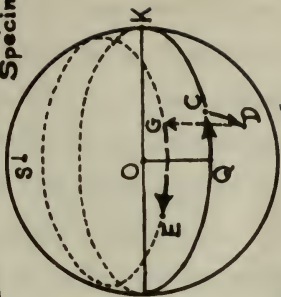
Ellipticity Compensator  
(Mica Plate)

b.  $C \rightarrow D$



d.  $Q \rightarrow C$

Opaque Specimen



$C. \left\{ \begin{array}{l} D \rightarrow G \text{ due to} \\ \text{retardation in} \\ \text{specimen. Same} \\ \text{reason for } G \rightarrow E \end{array} \right\}$

e.

$F \rightarrow Q$

d.  $E \rightarrow F$

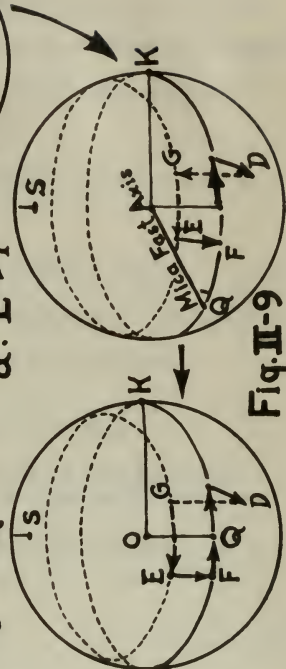


Fig. II-9





mica plate and Figure II-9(e) shows the rotation added when it passes through the quarter wave film again. This last point lies on the equator of the sphere indicating that it is plane polarized light (no latitude) and is in the same direction as the incident light (no change in longitude). Thus the ellipticity has been compensated for and when viewed through a crossed analyzer there should be extinction.

From Figure II-9, it can be shown by projection geometry (see Appendix A) that

$$\sin \Delta = \sin \phi \left[ \sin(2m + 2d_1) + \sin(2m - 2d_2) \right] \quad \text{--- (32)}$$

where  $\Delta$  is the phase shift on the sphere due to the specimen

$m$  is the angular displacement of the mica plate  
fast axis from zero

$\phi$  is the phase shift due to the mica plate

$d_1$  and  $d_2$  are individual rotations of incident and  
reflected beams due to rotation compensator.

Knowing that  $d_1$  and  $d_2$  are small and about equal and that  $\Delta$  is a small angle, we obtain

$$\Delta = 2 \sin \phi \sin 2m \quad \text{--- (33)}$$

where  $\Delta$  is in radians.

When the specimen is in the diagonal position, the incident light is reflected with maximum ellipticity. This elliptical light has components vibrating parallel and perpendicular to the direction of polarization. These two components will be out of phase since the light is elliptical. The phase angle between these components which is caused by the specimen is called  $\tau$ , the characteristic angle.

For each type of specimen there are many anisotropy parameters. These consist of special functions of the index of refraction, the coefficient of absorption, the ellipticity of the waves penetrating into the





specimen, and the index of the immersion medium. However, all of these functions are related to two of the parameters. One will be called the polarization coefficient  $\epsilon$  and the other the phase coefficient  $\delta$ . It has been shown that  $\epsilon$  is the tangent of the azimuth of the major axis of the reflected vibration ellipse measured from the vibration direction of the incident light (Eq. 30) and  $\delta$  is the ratio of the minor axis to the major axis. For incident linearly polarized light, the reflected ellipse can be written in the form

$$ve^{i\tau} = \epsilon + i\delta \quad \text{-----} \quad (34)$$

where  $v$  is the relative anisotropy and is equal to  $\sqrt{\epsilon^2 + \delta^2}$

$$\tan \tau = \frac{\delta}{\epsilon} \quad \text{-----} \quad (35)$$

on the sphere;  $\delta = \tan \omega \quad \text{-----} \quad (36)$

and  $\omega$  is one-half the latitude of a point on the sphere.

Since we are referring to the phase shift (change of latitude) due to the specimen (Figure II-9(c)).

$$-\delta = \tan \frac{\Delta}{2} \quad (\text{minus due to convention on sphere})$$

or for small angles of  $\Delta$

$$\Delta = -2\delta \quad \text{-----} \quad (36)$$

From Figure II-8 and Equation (30) it is seen that the tangent of angle between the major axis of the ellipse and the incident plane light in terms of the compensator is

$$\tan (\psi_d - \psi_e) = \epsilon = \frac{R^2 - 1}{2R} \sin 2r \quad (30)$$

D 52003



Substituting equations (56) and (50) into equation (35), gives

$$\tan \bar{\tau} = \frac{-\sin \phi \sin 2m}{\frac{R^2 - 1}{2R} \sin 2r} \quad - - - - (57)$$

However, the quarter-wave film in the rotation compensator is exact only for one wave length of light. For wave lengths longer or shorter than this, the light will suffer a relative phase change in passing through. Therefore equation (57) must be corrected for the wave length of light used and becomes

$$\tan \bar{\tau} = \frac{-\sin \phi \sin 2m + \sin \rho \sin 2r}{\frac{R^2 - 1}{2R} \sin 2r} \quad (58)$$

where the quantities  $\phi$ ,  $\rho$ , and  $R$  are determined experimentally and are constants of the compensator.

It should be noted in the above derivation that the  $\bar{\tau}$  used is not strictly the characteristic angle since the incident light is not plane but elliptical due to passing through the rotation and elliptical compensators once. However, for practical purposes, the approximation is not too bad.

The Bausch and Lomb Elliptical Vibration Compensator (Figure III-8) has calibrated divided circles upon which can be read the settings on the rotation and elliptical compensators. The readings for extinction on two successive diagonal positions of the specimen are subtracted to give the values of  $2m$  and  $2r$  to be used in equation (58).

$\bar{\tau}$  is a unique value — it is a constant for a particular anisotropic metal or numeral regardless of the angle of the basal plane or cleavage plane (provided that the surface condition is the same in each case). The value of  $\bar{\tau}$  for isotropic crystals is not constant and varies



between  $-90^{\circ}$  to  $+90^{\circ}$ . For anisotropic crystals  $\tau$  will show a change with surface characteristics which are undetectable under ordinary microscopic examination. The measurement of  $\tau$  serves as a criterion of the excellence of the polishing technique - the highest value of  $\tau$  being the best polished. Tabulated values showing this fact are available.<sup>9</sup>

If an increase in the characteristic angle,  $\tau$ , can be used as a criterion for the excellence of polishing techniques, then decreasing angles of  $\tau$  could serve to indicate a film growth on a polished surface. Therefore, the authors propose to study and attempt to determine the corrosion characteristics of zirconium based on theory described in this section.

D 52003



Chapter IIIPROCEDURE AND EQUIPMENTA. Preparation of Specimens

The original material from which all specimens were prepared was an 18" piece of Westinghouse Grade I Crystal Bar Zirconium, approximately 5/8" in diameter. The existing surface film was removed by wirebrushing and by chemical etching in a 1:1 solution of water and nitric acid plus a few drops of concentrated hydrofluoric acid. When all of the film had been removed, the bar was washed under tap water.

From this original bar, a four-inch billet (approximately equal to 100 grams) was cut from the center section. This billet was inspected again for any surface film, especially in and around the jagged grain boundaries of the surface crystals and then it was rewashed and dried in acetone.

In order to homogenize this "mother billet," it was drip melted twice under a vacuum of  $10^{-5}$  microns in a quartz tube using high frequency induction heating as shown in Fig. III-1. The quartz tube was evacuated by the usual laboratory fore pump, diffusion pump vacuum system. The zirconium billet was dripped into a standard M.I.T. copper crucible which is shown in cross section in Figure III-2. Previous work had shown that copper pickup from such a crucible was negligible. After each of the first few drip-meltings, the quartz tube (Figure III-1) showed a slight dark film deposit on the inside surface in the vicinity of the position where the zirconium had been suspended during the melt. This film is believed to be caused by condensation of the miscellaneous minor

D 52003

THE  
HISTORY OF THE  
CITY OF BOSTON

BY  
JOHN H. COOPER

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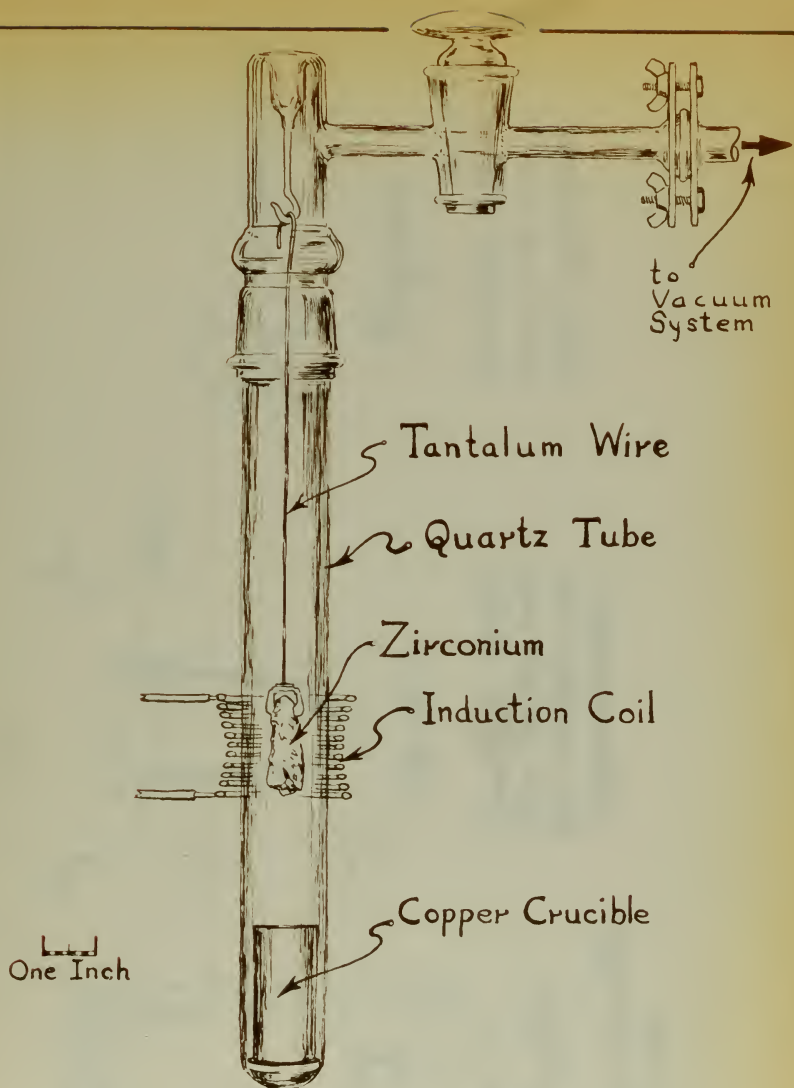
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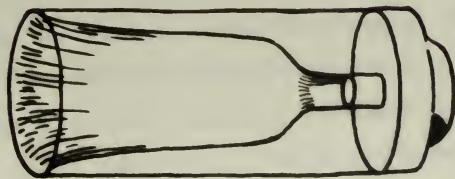




## VACUUM DRIP MELTING APPARATUS

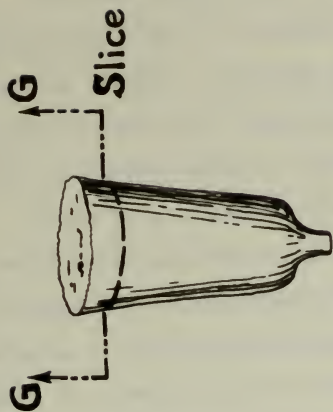
Fig - III - 1

00002 0



**Fig. III-2**

**Copper Crucible  
FULL SIZE**



**Fig. III-3**

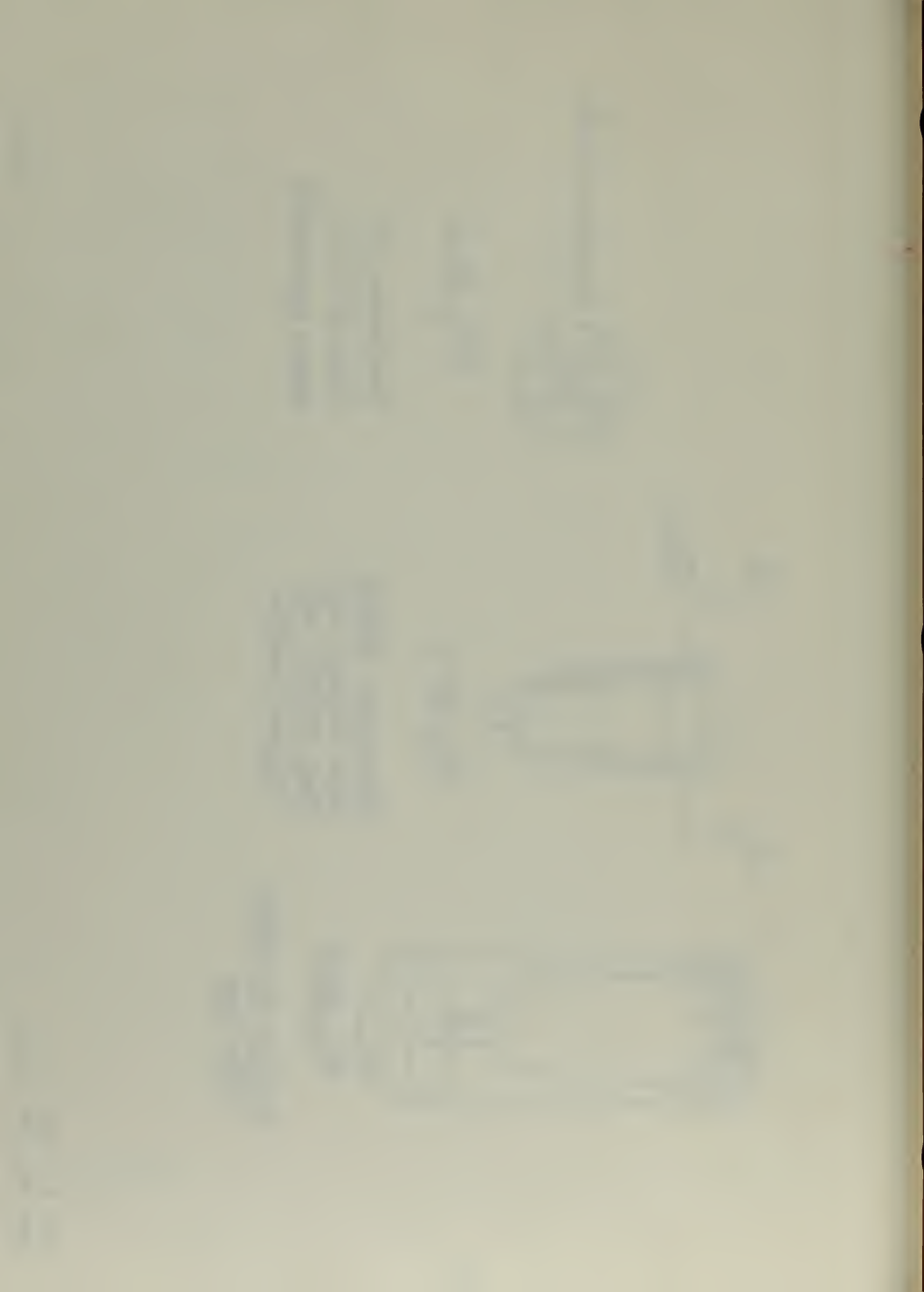
**Zirconium Billet  
after having been  
Drip Melted into  
Copper Crucible**



**Section G-G**

**Fig. III-4**

**Sectioning of  
Slice G-G to  
obtain specimen H**



impurities (e.g., Fe) volatilized during the melting process. It was noted that the amount of film deposited after each melt was less than for the previous melt. This suggested that the major percentage of the impurities of the "mother billet" were volatilized in the first few drip-melts.

Since, from previous experience, a vacuum of  $10^{-5}$  microns was set as the amount of evacuation necessary to avoid appreciable oxygen pickup during any high temperature process involving zirconium, the word "vacuum" throughout the rest of this paper will mean a vacuum of  $10^{-5}$  microns.

After the "mother billet" had been drip melted twice, it was held under vacuum at  $1600^{\circ}\text{C}$  for about ten minutes in order to further homogenize the material. The first specimen (piece H, Fig. III-4) then was roughed out from a  $1/8$ " thick slice cut off from the large diameter end of the drip melted billet as shown in Figures III-3 and III-4.

Piece (H) of Figure III-4 was prepared for metallographic examination while the remainder of the slice was given to the chemist for analysis. Upon completion of the chemical analysis, the material then remaining was subjected to a corrosion-resisting quality test in distilled water at  $315^{\circ}\text{C}$  for approximately 800 hours. Samples A, C, D, and F were so tested.

After the first slice had been cut from the main billet, nitrogen was added to the remainder of the billet. From this billet the second slice was cut yielding the second sample. This process was repeated until six samples had been prepared with increasing quantities of nitrogen. After each nitrogen addition, the billet was drip-melted in vacuum twice to provide homogeneity. A sample calculation to determine the pressure of nitrogen to be bled into the vacuum system in order to add a desired number of parts per million of nitrogen to the billet is given in Appendix B. The six samples so prepared with their nitrogen content in parts per

The first part of the paper discusses the importance of the study of the history of the English language. It is noted that the English language has a long and varied history, and that the study of its development is of great importance to the understanding of the language itself. The paper then goes on to discuss the various factors which have influenced the development of the English language, such as the influence of other languages, the influence of the social and cultural environment, and the influence of the individual writers and speakers. The paper concludes by stating that the study of the history of the English language is a fascinating and important field of study, and that it is one which should be pursued by all who are interested in the English language.

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million are chemically described in Table C-1 of Appendix C and were assigned the following symbols:

Specimen	ppm Nitrogen
A	6
B	70
C	113
D	147
E	150
F	240

The six specimens listed were prepared for metallographic examination as follows<sup>11</sup>:

- (a) Paper polished using kerosene on successively finer papers from 320 A down to 3/0 making each pass at 90° to the preceding pass.
- (b) Slightly etched in a solution of equal parts of water and nitric acid plus a few drops of hydrofluoric acid, washed in water and dried in acetone.
- (c) Electropolished in a solution of one part of 60% perchloric acid to ten parts of glacial acetic acid using a stainless steel anode and approximately 0.5 amperes and 18 volts for about 45 seconds. Experience and skill is needed in this phase in order to produce a "standard" electropolished surface, free from any macroscopic film and surface irregularities. Since a chemical etch film can be produced if the sample is left in the solution after the current is turned off, the sample always was withdrawn from the solution with the current still on. Moreover, since electropolishing for an



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excessively long period of time or with excessively high voltage can cause surface deviations, these conditions had to be avoided. Two of the samples inadvertently were excessively electropolished on the first try and had to be paper polished, etched and electropolished over again. All six samples when completed, however, showed a remarkably "standard" surface. The values of the characteristic angle for all of the anisotropic zirconium crystals selected did not differ by more than  $\pm 2$ . Moreover, after corroding these specimens fifteen times, the surfaces again were prepared in the same manner and gave reproducible results of the characteristic angle of the same crystals, within the same limit of error. After each electropolishing operation, the surface was washed with denatured ethyl alcohol and immediately hot-air dried.

#### B. Metallographic Procedure and Equipment

A detailed description of the equipment used is included in S.B. thesis, M.I.T., 1961, by E. L. Bronson.<sup>10</sup> A photograph of this equipment is shown in Figure III-7.

The general procedure followed was to use an Elliptical Vibration Compensator to measure the phase shift and rotation in the reflected polarized light from a particular portion of a selected crystal after successive growths of corrosion film. In order to do this, it was necessary to examine each specimen very carefully under polarized light in order to select one or more crystals in that specimen which had the following characteristics:

- (a) Large enough ( $1/32"$  x  $1/32"$  minimum).
- (b) Unique irregularities in shape of boundaries to permit easy identification.

The first of these is the fact that the  
government has been unable to  
obtain the necessary funds to  
carry out its policy. This is due  
to the fact that the government  
has been unable to raise the  
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has been unable to raise the  
necessary funds to carry out its  
policy. This is due to the fact  
that the government has been  
unable to raise the necessary  
funds to carry out its policy.

- (c) Free from spots or areas which became alternately bright and dark but not in phase sequence with the background crystal material.
- (d) Free from twin bands.
- (e) As far as possible, free from pits, inclusions, and spots which remained dark throughout an entire rotation.
- (f) As far as possible, free from shaded variations in color that appear within a crystal on some angles of rotation while appearing uniform in color on other angles. This "mottled effect" previously has shown up in drip-melted samples, but its nature and cause still remain unknown.

Two crystals were selected in each of samples A and F, while one crystal was selected in each of samples B, C, D, and E. The two crystals in A and F were chosen in order to determine whether the orientation of the basal planes had any effect on the variation in the phase shift and in the rotation of the plane of polarization from the surface of the randomly oriented crystals and from the corrosion films grown thereon.

Once the eight crystals were selected, they were photographed at 50 X in order to preserve a record of their contours to assist in identification after the corrosion films had been grown.

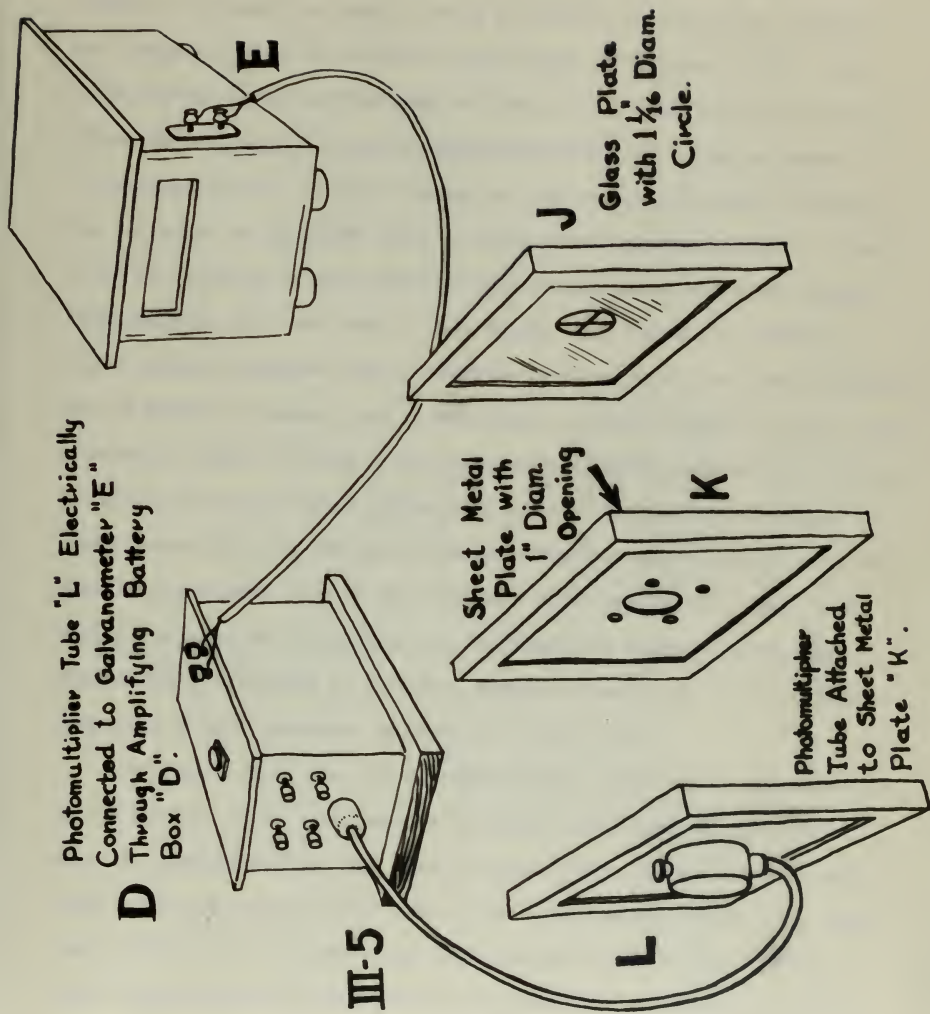
Having chosen a particular crystal in each sample, it was necessary to select a particular area in each sample and to devise a means which would permit observations of that particular area (and only that area) after each successive autoclaving. The equipment shown in Fig. III-5, pieces J, K, and L were designed to accomplish this function.

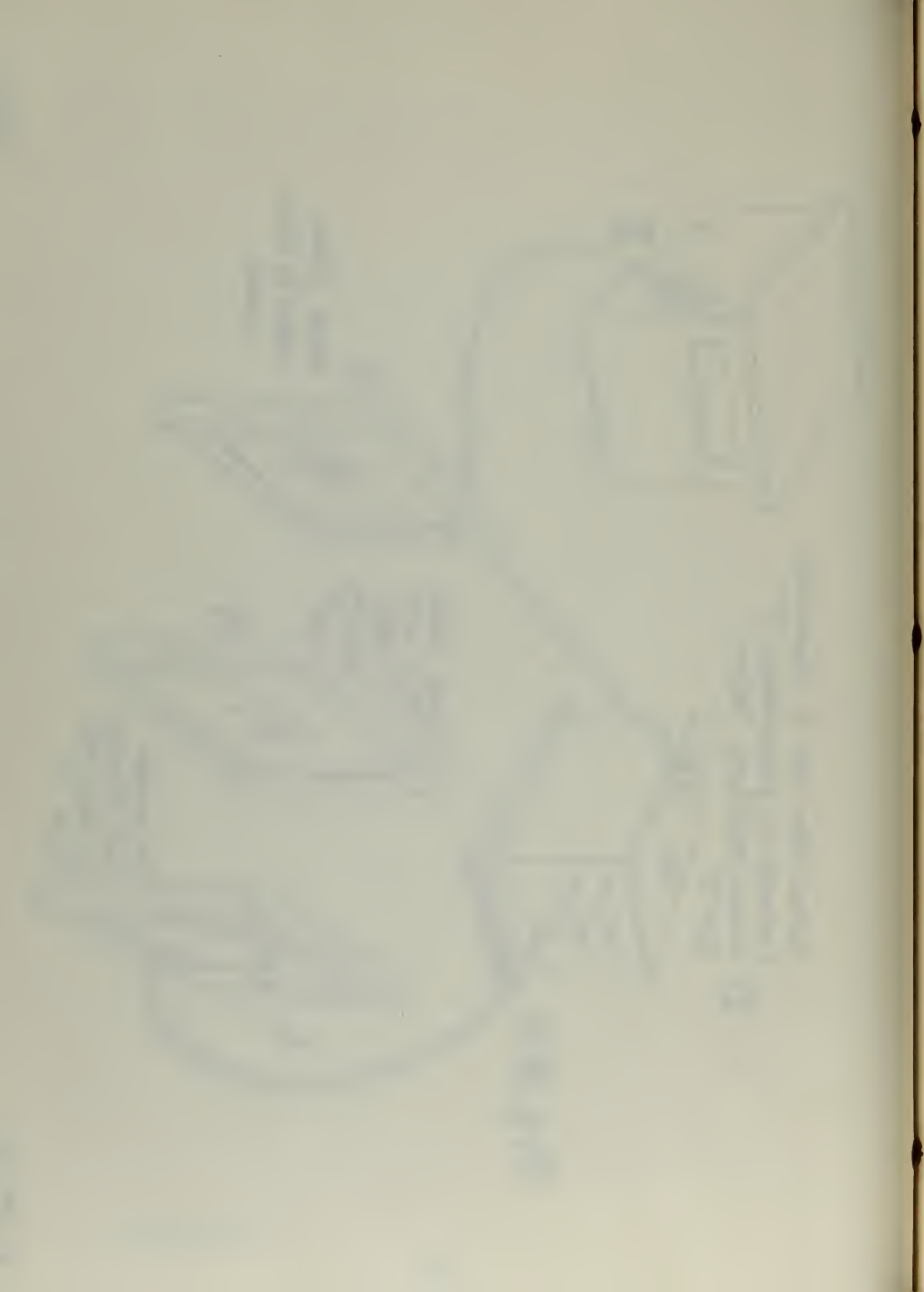
In Figure III-5, piece J is a frosted glass plate set in a metallograph photographic frame. A 1-1/16" diameter circle and perpendicular



Photomultiplier Tube "L" Electrically  
Connected to Galvanometer "E"  
Through Amplifying Battery  
Box "D".

Fig. III-5



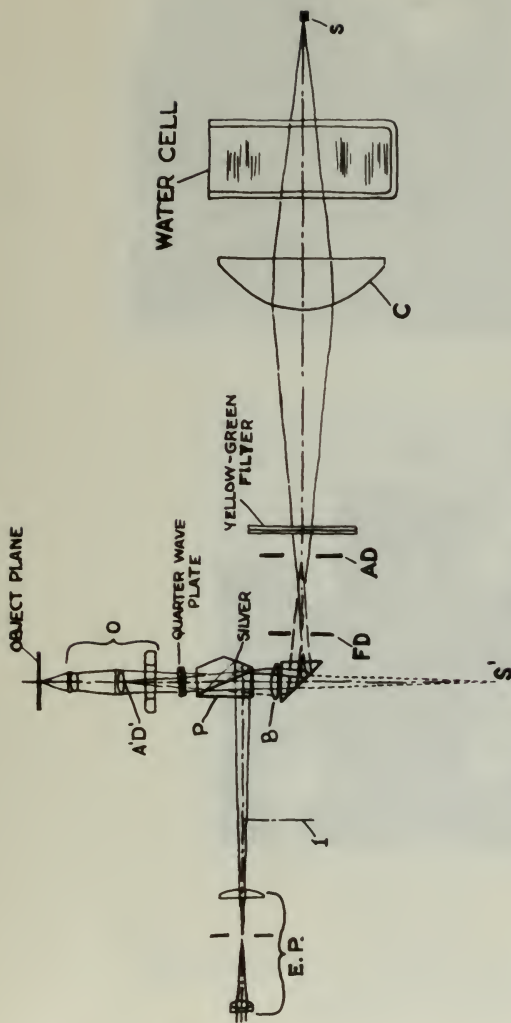




"cross hairs" were drawn in black ink at the center of the glass plate. Next a sheet metal plate with a one-inch circular hole cut out of its center was fitted into another frame so that by superposition, the hole was concentric with the 1-1/16" circle drawn on the glass plate. Four 5/32" bolting holes were drilled as shown in K of Figure III-5. To this plate the housing for the photomultiplier tube<sup>12</sup> was bolted as shown in L of Figure III-5. The metallograph was now ready to be used to measure the intensity of polarized light as reflected from the specimens. Figure III-6 is a diagram of the optical system in the Pausch and Lomb Research Metallograph, which was used in this study. The Carbon Arc source of light normally supplied with the metallograph previously had been replaced with a 40-watt Zirconarc tube which, after a warm-up period of three hours, provided a source of white light which was reasonably constant in intensity. To assist the Zirconarc in maintaining constant intensity, a Raytheon 100 power factor 120 volt 120 watts voltage stabilizer was connected into the alternating current feed to the Zirconarc Power Supply Box. This Power Supply Box acted as a converter and furnished the Zirconarc tube with approximately 2 amperes at 30 volts, direct current. All of the equipment described in this paragraph is shown in Figure III-7.

To make certain that the polarized light always struck perpendicular to the surface of the specimen (or its film), each specimen was viewed with its electropolished side down on a thin glass plate which had been made to fit the recess in the head of the metallograph. This glass plate had a 1/4" hole cut at the center with the edges of the hole carefully ground over so that no protruding sharp edges were available to scratch the surface to be studied.





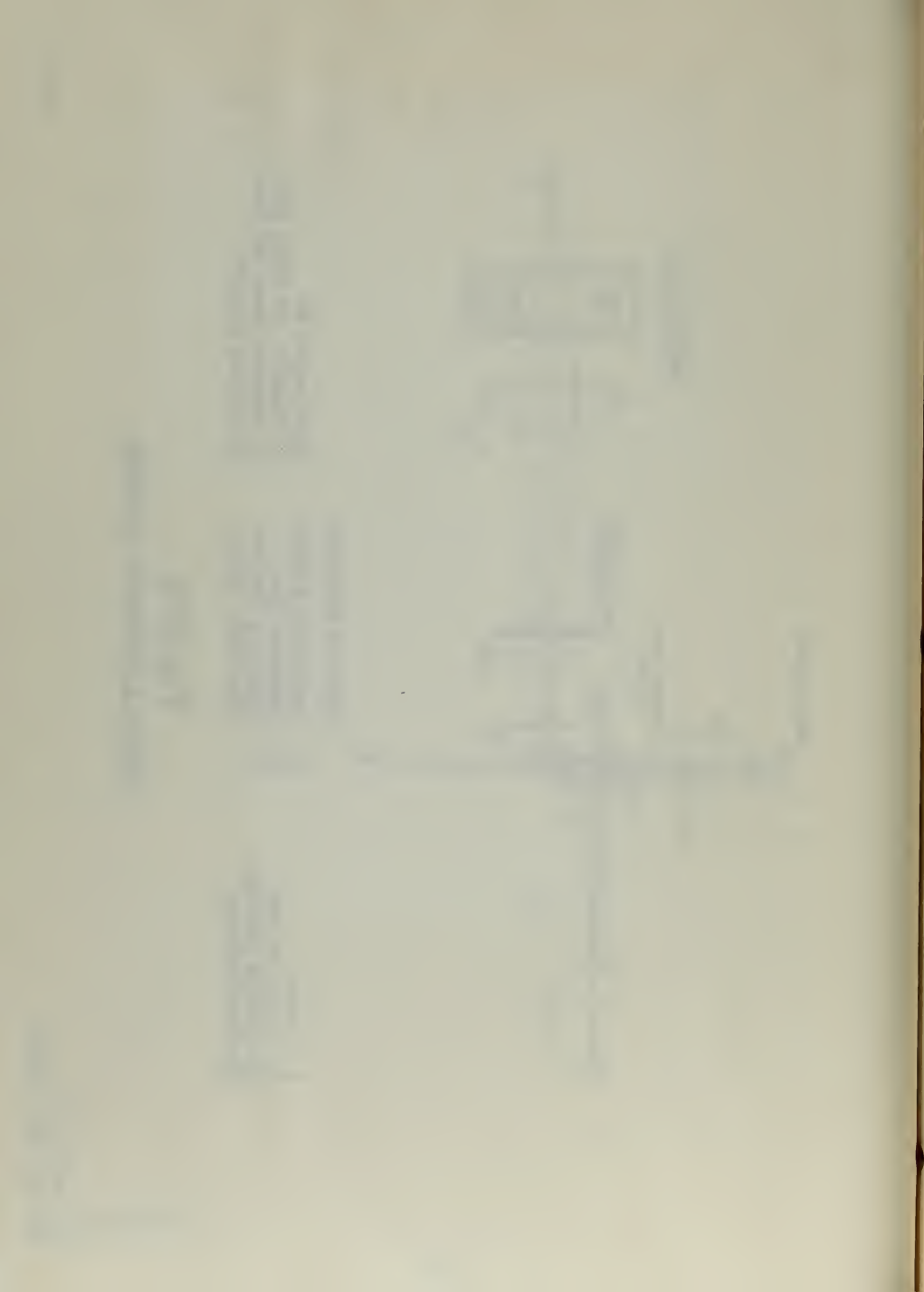
Bright-field Illumination

*S* - Source  
*C* - Condenser Lens  
*AD* - Aperture Diaphragm  
*FD* - Field Diaphragm

*S'* - Virtual Image of Source  
*B* - Condenser Lens  
*P* - Polarizing Vertical Illumination Prism

*A'D'* - Image of *AD*  
*O* - Objective and Glass Base  
*I* - Position of Swing-Out Prism  
*EP* - Camera Eyepiece.

**Fig. III-6**  
**METALLOGRAPH OPTICS**



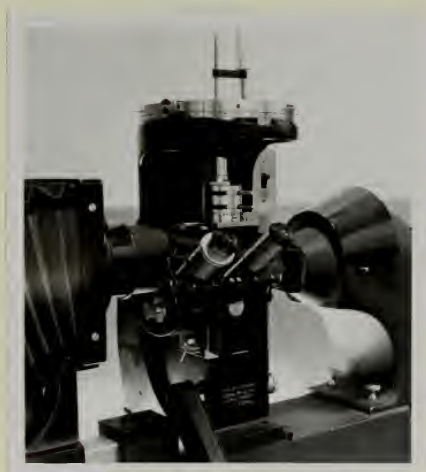


FIGURE III-7  
METALLOGRAPH AND ASSOCIATED EQUIPMENT WITH ELLIPTICAL  
COMPENSATOR IN PLACE

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FIGURE III-8  
ELLIPTICAL VIBRATION COMPENSATOR

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The specimen was then centered over the hole in the glass plate so that the metallograph beam fell on the desired crystal. This was most expeditiously accomplished by scanning the sample with a relatively low magnification (5.6 X) objective lens in the metallograph. Once the crystal was located, a 13.5 X objective lens was inserted in place of the 5.6 lens. With a combination of a 13.5 objective lens and a 10 X hyperplane eyepiece, and the bellows extended to 38.6 cm, the magnification obtained was 200 X. Once the cross hairs of the glass plate (J), Figure III-5, were located in a "clean" area of the crystal, a map of the exact location of the cross hairs and the 1-1/16" circle relative to the adjacent grain boundaries was traced on a transparent paper. With this map, it was relatively easy to reposition the crystal after each autoclaving so that the photomultiplier tube was measuring light intensity from the same area each time. It also was necessary to align the objective lens in the metallograph for each crystal so that upon rotation of the stage through  $360^{\circ}$  the crystal would revolve concentrically around the centering cross hairs. This alignment was most easily and most accurately accomplished by viewing the rotation at 200 X on the glass plate.

Once the crystal had been located accurately, the metallograph stage was rotated in increments of  $10^{\circ}$  through  $360^{\circ}$ . The amplification of the photomultiplier circuit<sup>12</sup> was adjusted at the battery box to give readings on the galvanometer ranging between 0 and 100. Instead of tabulating the readings, they were more rapidly plotted directly on reproducible graph paper forms printed especially for this purpose (Appendix D). This portion of the data was taken in order to locate the four azimuthal positions of maximum intensities of the reflected light during a full

D 52003



rotation. Henceforth these four positions will be called the diagonal positions of the crystal. The entire curve was recorded, instead of just the peaks, since this plot permitted a graphical location of the exact diagonal positions more accurately than could be obtained merely by sweeping with the galvanometer in the vicinity of the peaks. As it turned out, other interesting physical phenomena became apparent after studying and comparing the shape of the intensity curves.

Having determined the four angles of setting of the metallograph stage for the diagonal positions, the 13.5 objective lens was removed and the Elliptical Vibration Compensator was inserted in its place. The compensator was centered in the same manner as was the 13.5 lens and the exact area to be scanned again was located on the glass plate (J) Figure III-5. This was necessary because each time a lens was removed from the metallograph and then replaced, it was found that its optical axis would not return exactly to its previous position. Having centered the elliptical compensator and relocated the area on the sample with the aid of the tracing, the metallograph table was set on each of the azimuths of the diagonal positions at which points the elliptical compensator was adjusted for extinction. Figure III-8 is a photograph of the elliptical compensator and Figure II-8 is a sketch of its optical system. In Figure III-8 the upper pin controls phase shift while the lower pin controls rotation of the plane of polarization. To adjust the compensator for extinction both pins are rotated by trial and error until the galvanometer gives a minimum reading. The significance of this operation is described in Chapter II. Two extinction readings were taken at each of the four diagonal positions. The phase shift and rotation due to the specimen are included on each graphical form sheet in Appendix D.



### C. Autoclaving Procedure

Since the basic problem consisted of using polarized light to study the characteristics of the corrosion film on zirconium, it was important to be able to correlate the data obtained with some corrosion parameter. Previous work by the authors had shown that  $100^{\circ}\text{C}$  was too low and  $815^{\circ}\text{C}$  was too high for optimum growth of corrosion film for this type of study. Consequently, during run #1, successive autoclavings were carried out at a temperature of  $235^{\circ}\text{C}$  for equal increments of 15 minutes each. In run #2 the time of each autoclaving remained 15 minutes but the temperature was raised to  $295^{\circ}\text{C}$ .

The autoclaving for run #1 is described in detail as follows. The stainless steel autoclave was filled with distilled water and the six specimens were placed in the water with electropolished surfaces facing upwards. The cover was then bolted and the autoclave was heated in an electric oven from room temperature to  $235^{\circ}\text{C}$  using a constant setting on a Variac in the circuit. The time required to bring the autoclave up to temperature in this manner was approximately two hours and twenty-two minutes, and this time remained reasonably constant varying by not more than  $\pm 8$  minutes. As soon as the autoclave temperature reached  $233^{\circ}\text{C}$ , the autoclave immediately was transferred to a preheated oven with automatic controls which maintained the autoclave temperature at  $233^{\circ}\text{C}$ . After 15 minutes at this temperature, the autoclave was removed, sprayed with water until this sprayed water no longer turned to steam and then immersed in tap water. The entire cooling procedure took approximately five minutes and was constant to within  $\pm 1/2$  minute. Once cooled, the autoclave was opened, the samples removed and air-dried with a syringe. For each successive autoclaving, new distilled water was used. Calibrated Chromel-Alumel thermocouples correct to  $\pm 1^{\circ}\text{C}$  were used.

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Run #2 was conducted in a manner similar to run #1 except that only one autoclaving was done and its temperature was 295° C. The time required to bring the autoclave up to 295° C was 2 hours and 53 minutes. The cooling time was 5 minutes.

In run #1, the corrosion film varied in color from a pale yellow after the first autoclaving, on through straw, to medium brown to bluish for the last autoclaving. In general the changes in film color were similar to those obtained in tempering steel. Polarized light readings were obtained up through the bluish films.

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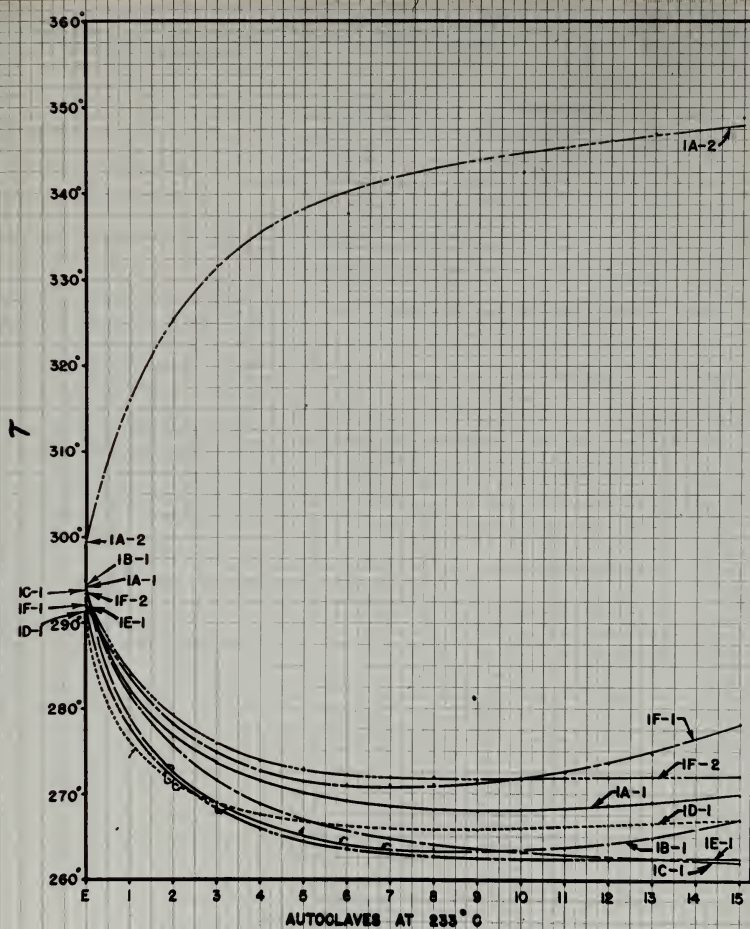


Chapter IVRESULTS AND CONCLUSIONS

1. The values obtained for either  $2m$ ,  $2r$ , or  $\bar{C}$  do not vary in any manner consistent with the nitrogen content in the specimens. Therefore, at present this method does not appear suitable for use as a corrosion-resistance test for zirconium.
2. With further refinement in methods of sample preparation and with more accurate optical systems, it still may be possible to use polarized light to determine corrosion-resistance of zirconium. The details of what refinements are necessary and what optical errors should be eliminated are included in Chapter V.
3. Three possible explanations of why the value of  $\bar{C}$  did not vary as the nitrogen content in the specimens are as follows:
  - A. As shown in Appendix C, the inadvertent impurities in the specimens were considerable compared to nitrogen content. For example, in the low nitrogen sample 1A-1 there was about twenty times as much iron and carbon as there was nitrogen. In the remainder of the samples, as the content of nitrogen was increased, volatilization reduced the quantities of other impurities. It is entirely possible that the variation of the effects on corrosion of these other impurities was greater than that of the nitrogen.
  - B. Some metals corrode at different rates along different directions. Zirconium, being anisotropic in other properties, probably is also anisotropic in corrosion. Therefore, with random orientation of the eight crystals studied, the effect of variable corrosion



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SUMMARY PLOT OF CHARACTERISTIC  
ANGLES ( $\gamma$ ) FOR RUN NO. 1

Fig IV-1

NET NET LAP  
PLOT JEN HEN GRAPHIC

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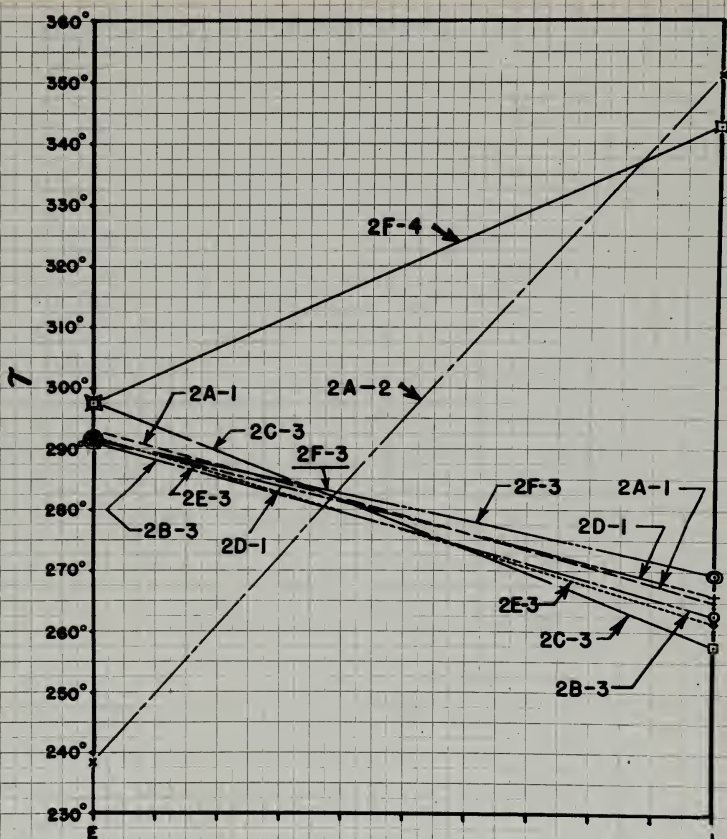
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AUTOCLAVES AT 295°C

SUMMARY PLOT OF CHARACTERISTIC  
ANGLES ( $\gamma$ ) FOR RUN #2

MIT MET LAB  
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Fig IV-2

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growth caused by grain orientation may have been of greater magnitude than the effect of nitrogen content.

C. A combination of (A) and (B) acting together.

4. The anomalous behavior of curves 1A-2, 2A-2 and 2F-4 are explained by the fact that their optical axes were nearly perpendicular to the surface of the specimen. This statement is based on the fact that the specimens, when rotated upon the stage did not go through sharp maxima and minima of intensity, but instead remained fairly dark throughout the entire rotation. Since a hexagonal crystal has two of its three crystal axes of equal magnitude and since these two equal axes were contained in the surface of the specimen, there was very little ellipticity introduced into the incident plane polarized light. Consequently, the reflected polarized light contained practically no ellipticity which resulted in practically no phase shift. The rotation of the plane of polarization, however, was considerably more than for the other specimens. Hence with essentially a constant phase shift and an increasing rotation of the plane of polarization, the characteristic angle  $\tilde{U}$ , which depends on the ratio of the sines of these two parameters, increased rapidly as shown in Figures IV-1 and IV-2.
5. As the corrosion time was increased, the color of the specimens progressed from natural to straw, to yellow, to brown, to blue in overall appearance.
6. Looking at each specimen in more detail, the color of the film was not homogeneous across the surface of any specimen. For example, in a specimen where the overall appearance of the surface gave a brownish appearance, upon more detailed observation some areas



appeared yellow and some blue, whereas the majority appeared brown. The boundaries between these changes in color were sharp and had a general appearance similar to the boundaries of large regular zirconium crystals.

7. Additional zirconium crystal bar samples (not the A to F series discussed in this thesis) were corroded for eighty hours at  $315^{\circ}\text{C}$  plus six days at  $345^{\circ}\text{C}$ . A small amount of white powderish film became noticeable. This film was concentrated in white lines which appeared to be outlining zirconium crystal boundaries.
8. After the 15th autoclaving at  $233^{\circ}\text{F}$  (end of Run #1) the specimens were autoclaved at  $233^{\circ}\text{C}$  for about 40 hours. Upon metallographic examination the film was studded with reddish irregular spots which shone through the much more darkly colored background of the crystal. In addition, there were sets of longitudinal lines which appeared as cracks in the film. In this condition, the diagonal positions of maximum intensities could not accurately be determined, nor could the adjustments be made for minimum intensities on the elliptical compensator. Since the surface being studied was no longer homogeneous, these results were taken as being random and were discarded. From this experience it is felt that polarized light cannot be used to obtain quantitative effects on film growth after the film has either broken down in local areas, or split, or grown too thick.
9. The crystal boundaries remained the same shape and size when observed throughout the entire growth range of film growth. This fact indicates that the film was thin or that its structure was similar to the parent crystal structure.



10. Upon calibration of Elliptical Vibration Compensator #37 with a zirconarc source of white light, the new constants obtained are contained in the following corrected formula for the characteristic angle,  $\tau_p$

$$\tan \tau = -.6666 \frac{\sin 2m}{\sin 2r} + .0208$$

11. When specimens 1A-2, 2A-2 and 2F-4, (which had their optical axes almost normal to the surface), were rotated through  $360^\circ$ , only two points of maximum intensity appeared. Upon investigation, it was found that the 13.5 objective lens was strained. When another 13.5 objective lens was used, the usual four points of maximum intensity were observed. The diagonal positions of the remainder of the samples were checked using both 13.5 lenses and the azimuths coincided, showing no error in this source of the data.
12. The results of corroding specimens A, C, D and F in distilled water at  $315^\circ \text{C}$  for 800 hours showed that specimens A, C, and D were of about equal quality while specimen F showed greater corrosion.





Chapter VRECOMMENDATIONS FOR FURTHER INVESTIGATION

It is recommended that further study, using polarized light, be made of the characteristics of the corrosion film of zirconium. Some suggestions which may prove beneficial in such a study are listed as follows:

1. Preparation of specimens

- (a) Do all drip-melting in a vacuum of  $10^{-6}$  microns to reduce oxygen pickup in the specimens.
- (b) Since chemical analysis indicated that the largest percentages of miscellaneous impurities were removed by volatilization during the first few meltings, drip melt the mother billet of crystal bar about six times for reduction of impurities and for homogenization. Cut the billet into four parts and add nitrogen so that the specimens to be studied will contain approximately 0, 100, 200 and 300 parts per million of nitrogen. This procedure will give each specimen the same number of drip meltings and will reduce the variation in impurities.
- (c) Make specimens larger and provide an additional 10 grams for chemical analysis plus 5 grams for standard corrosion resistance test.

2. Since the metallograph optical system and its associated lenses are being returned to the factory for correction of optical errors, this problem should not be a factor in future work. However, periodic calibration checks should be made on the metallograph.
3. Instead of using a large single crystal in the specimen for study, it

D 52003



is suggested that the specimens consist of a large number of small grains that are given a preferential direction by cold working. By using a large area with small grains, i.e., about 100 grains, the problem may be approached in a statistical manner. This may be done by scribing an area on the specimen and centering this area after each autoclave so that the same area will be observed each time. Additional advantages of this method are that the effects of any preferential corrosion along grain boundaries will be included and any preferential corrosion due to crystal orientation will be eliminated. After any cold working, the specimens should be annealed. Twins in the field being measured give erroneous results.

4. Use the elliptical compensator with an optically inactive standard for locating the diagonal positions as well as for determining the phase shift and rotation in the reflected light. This procedure is outlined in the Bausch and Lomb Elliptical Vibration Compensator Instruction Book.<sup>9</sup>
5. Record intensities of diagonal positions and of minimum positions. In order to do this it will be desirable to make one or more "intensity standards" to which the galvanometer can be calibrated.
6. Autoclave at 295° C for one hour intervals.
7. Carefully note and record the color and general macroscopic surface appearance for each measurement. It is entirely possible that the corrosion time at which a specimen first shows film breakdown (as described in Chapt. IV-8) may be used as a corrosion parameter.



Appendix AGeometrical Analysis of Poincare Sphere For PhaseShift Due to Specimen

From Figure A-1

$$AB = OA \sin(2\alpha + 2d_1)$$

$$AC = AB \tan \phi_1 = OA \tan \phi_1 \sin(2\alpha + 2d_1)$$

$$\tan \delta_1 = \frac{AC}{OA} = \frac{OA \tan \phi_1 \sin(2\alpha + 2d_1)}{OA}$$

$$\tan \delta_1 = \tan \phi_1 \sin(2\alpha + 2d_1) \quad \text{----- (a)}$$

Similarly

$$PR = OP \sin(2\alpha - 2d_2)$$

$$QP = PR \tan \phi_2 = OP \tan \phi_2 \sin(2\alpha - 2d_2)$$

$$\tan \delta_2 = \frac{QP}{OP} = \frac{OP \tan \phi_2 \sin(2\alpha - 2d_2)}{OP}$$

$$\tan \delta_2 = \tan \phi_2 \sin(2\alpha - 2d_2) \quad \text{----- (b)}$$

Since phase shift due to specimen is  $\Delta = \delta_1 + \delta_2$

$$\text{and } \phi_1 \approx \phi_2 = \phi$$

adding (a) and (b)

$$\tan \delta_1 + \tan \delta_2 = \tan \phi \left[ \sin(2\alpha + 2d_1) + \sin(2\alpha - 2d_2) \right]$$

$$\text{since } \tan(\delta_1 + \delta_2) = \frac{\tan \delta_1 + \tan \delta_2}{1 - \tan \delta_1 \tan \delta_2}$$

$$\text{and since } \delta_1 \text{ and } \delta_2 \ll 1$$

$$\tan(\delta_1 + \delta_2) \approx \tan \delta_1 + \tan \delta_2$$

or

$$\tan \Delta = \tan \phi \left[ \sin(2\alpha + 2d_1) + \sin(2\alpha - 2d_2) \right]$$

and  $\Delta$  and  $\phi$  are both small so

$$\sin \Delta = \sin \phi \left[ \sin(2\alpha + 2d_1) + \sin(2\alpha - 2d_2) \right]$$



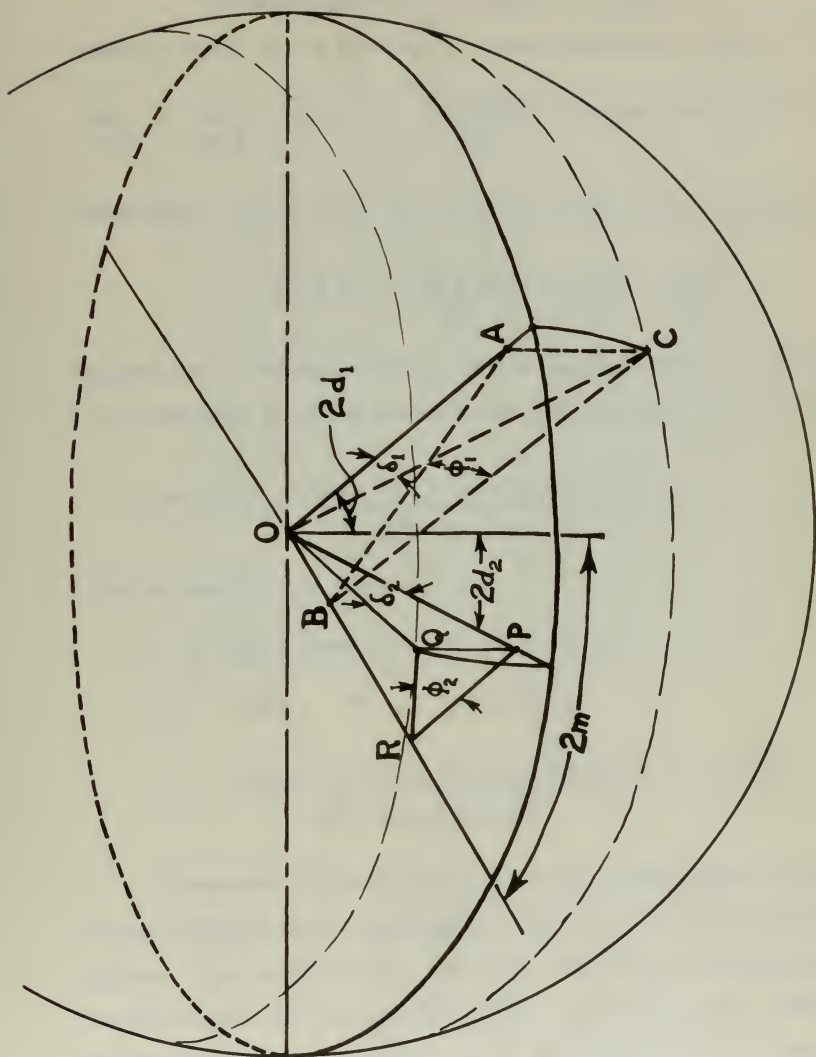


Fig. A-1





Appendix BSAMPLE CALCULATION FOR ADDITION OF NITROGEN

Problem: To add 40 ppm nitrogen to a zirconium billet weighing 87.5 gms.

$$\frac{40}{10^6} = \frac{x}{87.5} \quad \text{and} \quad x = \frac{40 \times 87.5}{10^6} = \text{number gms } N_2 \text{ needed}$$

Convert gms. of  $N_2$  to cc of  $N_2$  at STP and convert for room temperature

$$\text{cc of } N_2 = \frac{40 \times 87.5}{10^6} \times \frac{22,400}{28} \times \frac{300}{273}$$

Estimate that an excess of 20%  $N_2$  must be supplied because a portion of the available  $N_2$  is not absorbed when the zirconium billet is heated.

$$\therefore \text{cc of } N_2 = \frac{1.2 \times 40 \times 87.5 \times 22,400 \times 300}{10^6 \times 28 \times 273} = V_1$$

$$\text{Volume of manifold} = 325 \text{ cc} = V_2$$

$$P_1 = \text{atmos. press. (on this day} = 751 \text{ mm)}$$

$$P_1 V_1 = P_2 V_2 \quad \text{or} \quad P_2 = \frac{P_1 V_1}{V_2}$$

$$\therefore P_2 = \frac{751 \times 1.2 \times 40 \times 87.5 \times 22,400 \times 300}{10^6 \times 28 \times 273 \times 325} = 8.54 \text{ mm}$$

Consequently, bleed nitrogen from the circular flask (shown at the top of Fig. B-1) into the manifold until the pressure manometer reads 8.54 mm. Open the stopcock to the quartz tube (Fig. III-1) containing the zirconium billet and heat the billet by induction coil to  $1600^\circ \text{C}$  for about ten minutes or until the 8.54 mm pressure of  $N_2$  has been reduced essentially to the original vacuum pressure.



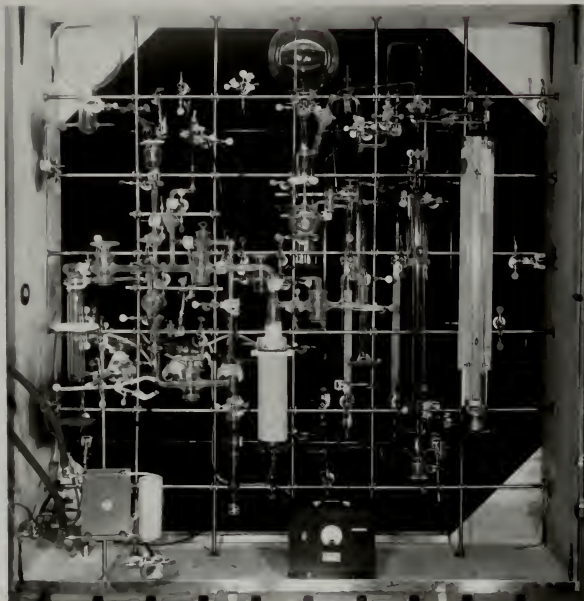


Figure B-1

TYPICAL LABORATORY APPARATUS FOR  
ADDING NITROGEN TO ZIRCONIUM



Appendix CCHEMICAL AND SPECTROGRAPHIC ANALYSIS

Table I gives the results of Chemical and Spectrographic Analysis of the original Westinghouse Grade I Crystal Bar Zirconium and of the six samples (A, B, C, D, E, and F) made from that material. During the nitrogen addition there was no substantial change in concentration of any other elements except oxygen, carbon and iron. Of these, the oxygen increased while the iron decreased. While the small amount of oxygen pickup noticed would not essentially alter one way or other the corrosion resisting qualities of the samples, the reduction in iron content might well have a pronounced effect. In particular, Sample A which was purest in nitrogen was most impure in iron, with a ratio of iron impurity to nitrogen impurity of more than 20:1.





TABLE C-I

CHEMICAL CONTENT OF PARENT ZIRCONIUM AND  
OF SIX SAMPLES MADE THEREFROM

	Chem. H Spect.	Chem. A Spect.	Chem. B Spect.	Chem. C Spect.	Chem. D Spect.	Chem. E Spect.	Chem. F Spect.
C	110	90		140	50	140	80
O	70	90	260	360	290	450	320
N	2	6	70	115	147	150	240
Fe	132						65
Si	<10-20	30	35	30	20		15
Al	54-40	45	40	40	30	40	55
Ti	<5-9	6	$\mu 1$	$\mu 1$	$\mu 1$	$\mu 1$	<5 $\mu 1$
Ni	6-3	5	$\mu 1$	$\mu 1$	$\mu 1$	$\mu 1$	<5 $\mu 1$
Cr	<5-65	30	25	20	25	30	<5 20
Ca	12	7	9	8	8	10	3
Cu	9	5	4	$\mu 1$	$\mu 1$	2	$\mu 1$
Mg	2	$\mu 1$	2	1	$\mu 1$	20	5
Mn	4	$\mu 1$	$\mu 1$	$\mu 1$		$\mu 1$	$\mu 1$
Mo	$\mu 10$	$\mu 10$	$\mu 10$	$\mu 10$	$\mu 10$	$\mu 10$	$\mu 10$
Pb	3	$\mu 1$	$\mu 1$	$\mu 1$	$\mu 1$	$\mu 1$	$\mu 1$
Sn	2	1	$\mu 1$	$\mu 1$	$\mu 1$		
V	$\mu 50$	$\mu 50$	$\mu 50$	$\mu 50$	$\mu 50$	$\mu 50$	$\mu 50$

D 52003



Appendix DData

The data consists of values of phase shift and of rotation of plane of polarization measured after electropolishing and after each autoclaving. It was plotted directly on graph paper forms designed especially for this purpose.

For each specimen for each run, a curve was faired through the measured values of  $2m$  and of  $2r$  versus the number of autoclavings. These curves are included at the end of each section of data. The values used in the calculations of Appendix F are values picked off the faired curves of data.



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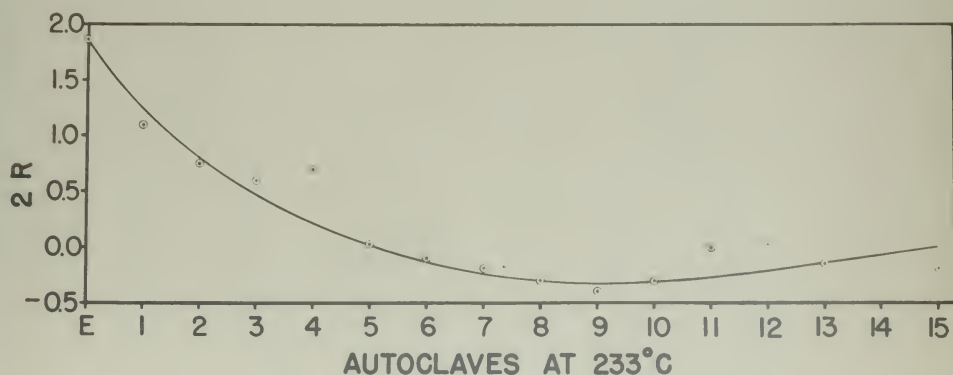
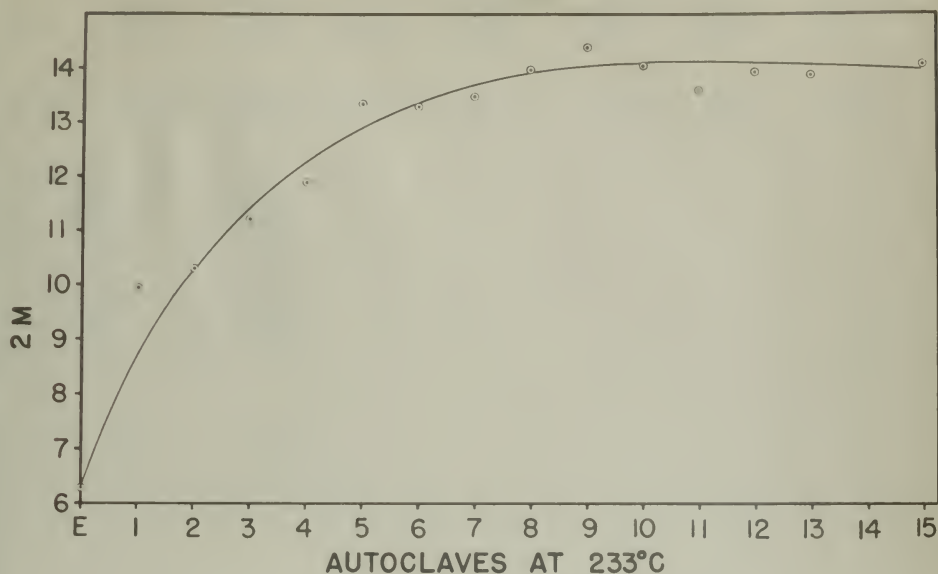
Run #1

Corrosion film was grown at 233° C

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PHASE SHIFT (2m) AND ROTATION OF PLANE OF  
POLARIZATION (2r) VS. CORROSION  
TIME FOR SAMPLE NO. 1A-1





1

CRYSTAL N

REFLECTOR CONDITION

AS ELECTROLYTICALLY POLISHED

080		170		260		350	
M <sub>1</sub>	M <sub>2</sub>	M <sub>1</sub>	M <sub>2</sub>	M <sub>1</sub>	M <sub>2</sub>	M <sub>1</sub>	M <sub>2</sub>
45.2	46.8	38.9	44.6	45.5	46.2	39.0	44.7
45.3	46.7	39.3	44.5	45.0	46.7	38.6	45.1
AV 45.25	46.75	39.10	44.55	45.25	46.45	38.80	44.90

M	45.25	R	46.75
M	39.10	R	44.55
$\Delta M$	6.15	$\Delta R$	2.20

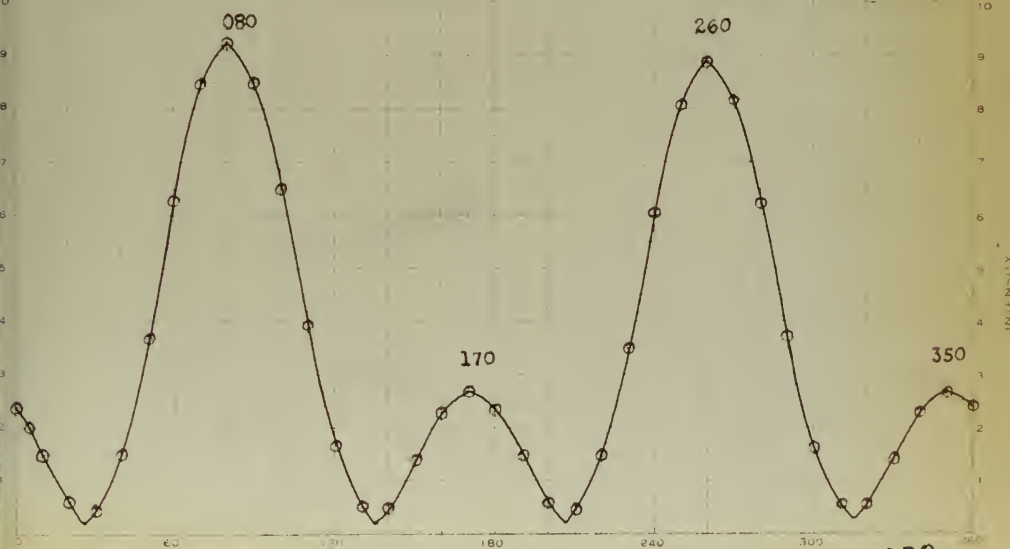
M	45.25	R	46.45
M	38.80	R	44.90
$\Delta M$	6.45	$\Delta R$	1.55

 AV  $\Delta M$  6.30 PHASE SHIFT

 AV  $\Delta R$  1.87 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



SAMPLE NO 1A

0 PPM N2

DATE 12 March PM

CRYSTAL NO 1

SURFACE FILM CONDITION

1st AUTOCLAVE 15 min 233°C

④ 332		④ 062		④ 152		④ 242	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.1	46.1	37.3	45.1	47.0	46.2	37.3	45.4
47.6	46.4	37.0	45.0	47.0	46.1	37.3	44.9
47.35	46.25	37.15	45.05	47.00	46.15	37.30	45.15

M <sub>1</sub>	47.35	R <sub>1</sub>	46.25
M <sub>2</sub>	37.15	R <sub>2</sub>	45.05
ΔM <sub>12</sub>	10.20	ΔR <sub>12</sub>	1.20

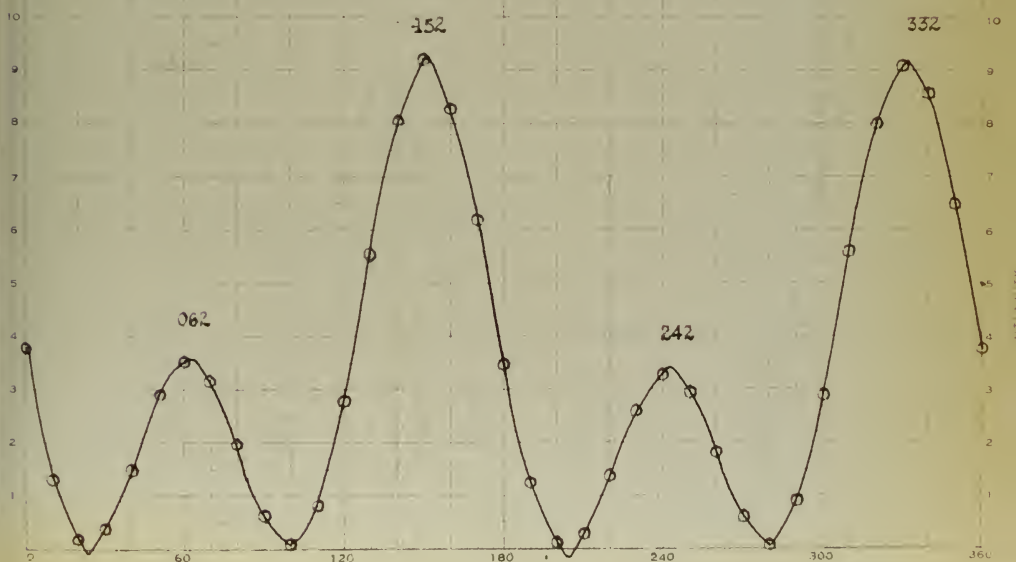
M <sub>3</sub>	47.00	R <sub>3</sub>	46.15
M <sub>4</sub>	37.30	R <sub>4</sub>	45.15
ΔM <sub>34</sub>	9.70	ΔR <sub>34</sub>	1.00

AV ΔM 9.05 PHASE SHIFT

AV ΔR 1.10 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



2nd AUTOCLAVE OF 15 Min. at 233°C

CRISTAL NO 1 SURFACE FILM CONDITION

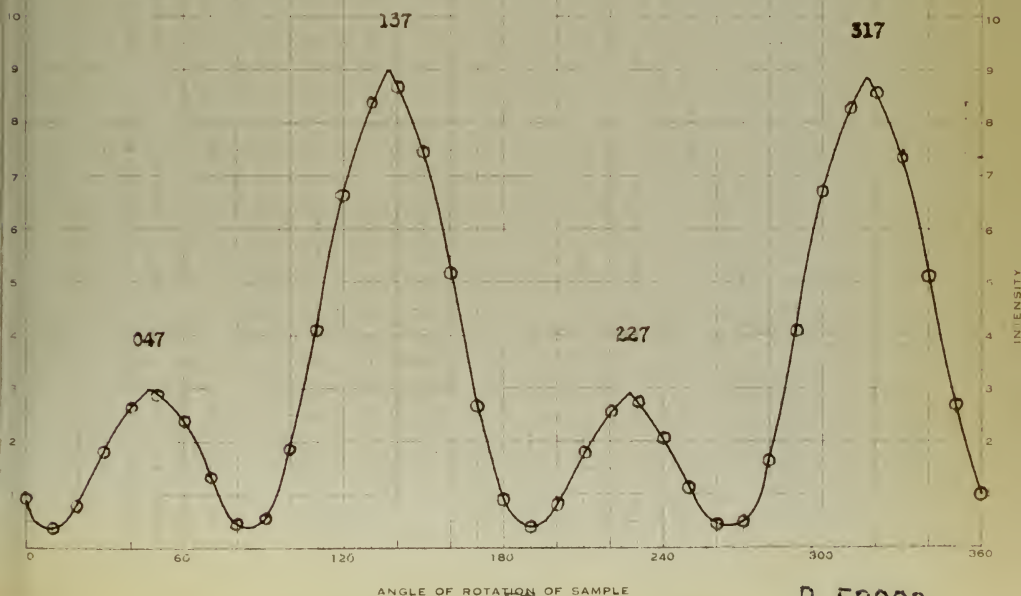
① 317		② 047		③ 137		④ 227	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.2	45.7	37.2	44.3	47.9	45.9	37.1	44.7
46.9	45.9	37.4	45.1	47.9	45.8	37.0	45.2
47.05	45.80	37.30	45.20	47.90	45.85	37.05	44.95

M	47.05	R	45.80
M	37.30	R <sub>2</sub>	45.20
Δ M	9.75	Δ R	.60

M	47.90	R	45.85
M <sub>4</sub>	37.05	R <sub>4</sub>	44.95
Δ M <sub>34</sub>	10.85	Δ R <sub>34</sub>	.90

AV Δ M 10.30 PHASE SHIFT  
 AV Δ R .75 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







CRYSTAL NO

SURFACE FILM CONDITION

AV

@ 144		@ 153		@ 148		@ 1	
M1	P	M2	R2	M3	R3	M4	R4
47.2	40.1	49.0	45.7	47.2	44.1	47.4	45.0
47.1	40.1	49.0	45.7	47.1	44.1	47.5	45.7
47.30	40.8	49.15	45.80	47.30	44.12	47.50	45.80

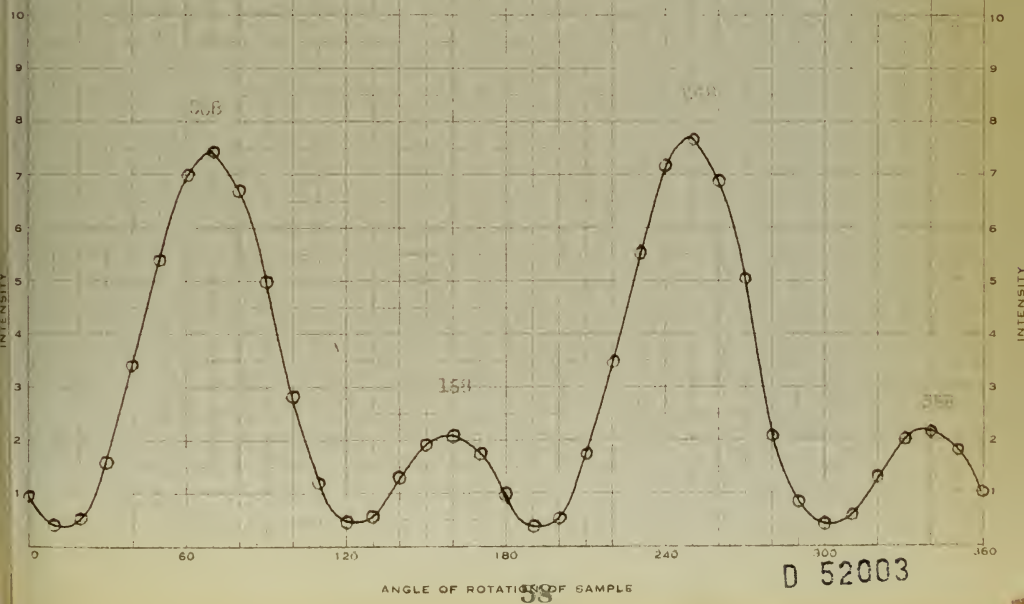
M1	47.30	R1	45.77
M2	49.10	R2	45.60
$\Delta M_{12}$	1.80	$\Delta R_{12}$	.87

M3	47.10	R3	44.15
M4	47.50	R4	45.70
$\Delta M_{34}$	0.40	$\Delta R_{34}$	.55

AV  $\Delta M$  11.80 PHASE SHIFT  
AV  $\Delta R$  .73 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CRYSTAL NO

SURFACE FILM CONDITION

①		②		③		④	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.7	45.5	46.1	44.4	47.	46.7	47.7	46.1
45.4	45.5	46.3	44.5	47.3	46.5	47.7	46.1
Av. 45.1	45.1	46.2	44.5	47.0	46.7	47.5	46.1

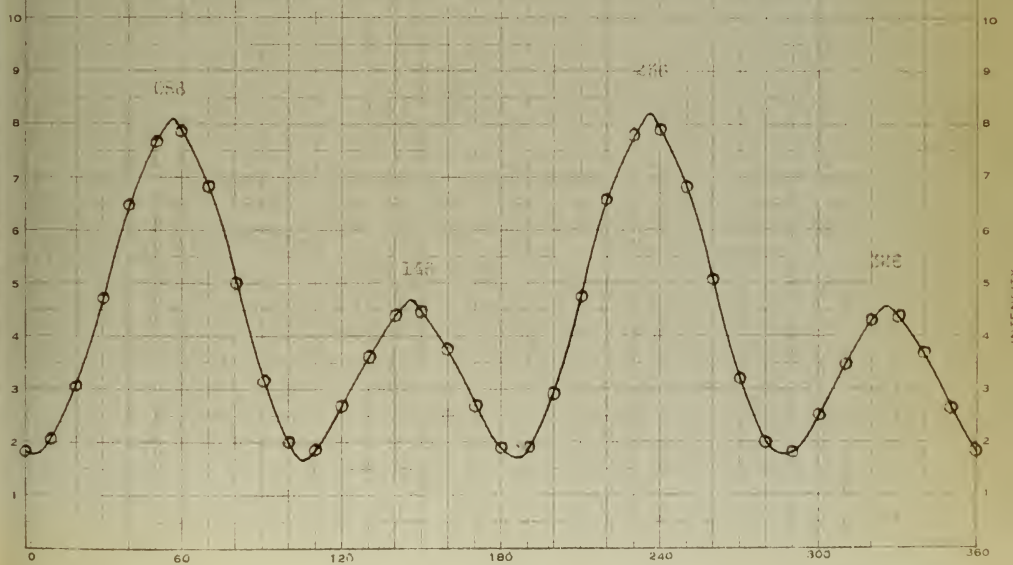
M <sub>1</sub>	45.1	R <sub>1</sub>	46.3
M <sub>2</sub>	45.5	R <sub>2</sub>	44.5
$\Delta M_{12}$	0.4	$\Delta R_{12}$	1.8

M <sub>3</sub>	47.0	R <sub>3</sub>	46.7
M <sub>4</sub>	47.5	R <sub>4</sub>	46.1
$\Delta M_{34}$	0.5	$\Delta R_{34}$	0.6

Av  $\Delta M$  11.78 PHASE SHIFTAv  $\Delta R$  .70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



CRYSTAL No. SURFACE FILM CONDITION

(i) 259		(ii) 275		(iii) 135		(iv) 255	
M	R	M	R	M	R	M	R
48.5	4.5	47.5	4.5	47.5	4.5	47.5	4.5
47.5	4.5	47.5	4.5	47.5	4.5	47.5	4.5
Av. 47.5	4.5	47.5	4.5	47.5	4.5	47.5	4.5

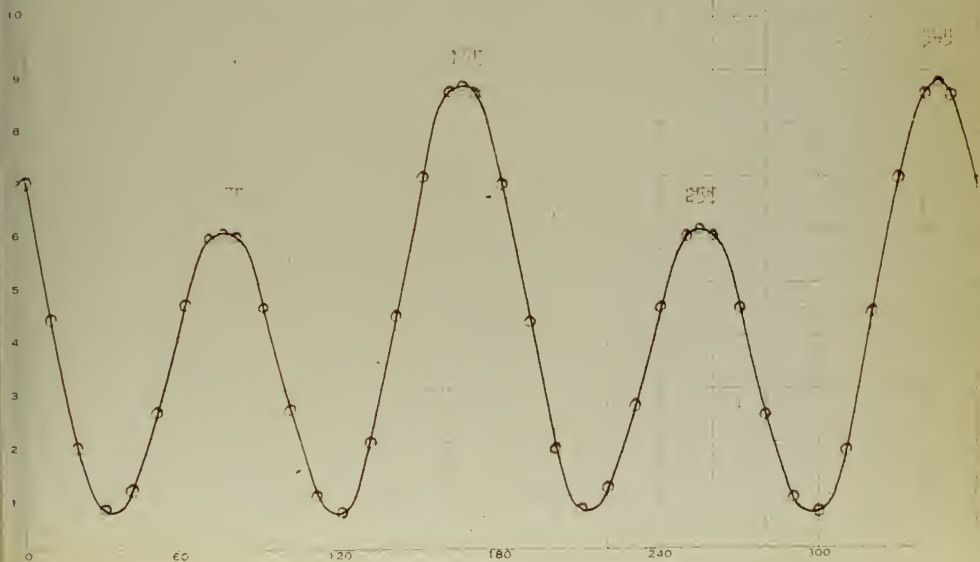
M 47.55 R 45.55  
M 47.55 R 45.55  
 $\Delta M$  17.80  $\Delta R$  0

M 47.55 R 45.55  
M 47.55 R 45.55  
 $\Delta M$  17.80  $\Delta R$  0

Av  $\Delta M$  17.80 PHASE SHIFT  
Av  $\Delta R$  0 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



CRYSTAL NO. 114

SURFACE FILM CONDITION

114		114		114		114	
M1	R	M2	R	M3	R	M4	R4
35.4	45.0	35.7	45.4	35.4	45.7	35.4	45.5
45.0	45.5	35.5	45.5	45.4	45.5	35.2	45.3
35.20	45.30	35.25	45.35	45.45	45.35	35.25	45.3

M	35.20	R	45.30
M <sub>1</sub>	35.25	R <sub>1</sub>	45.35
ΔM	13.35	ΔR	- .15

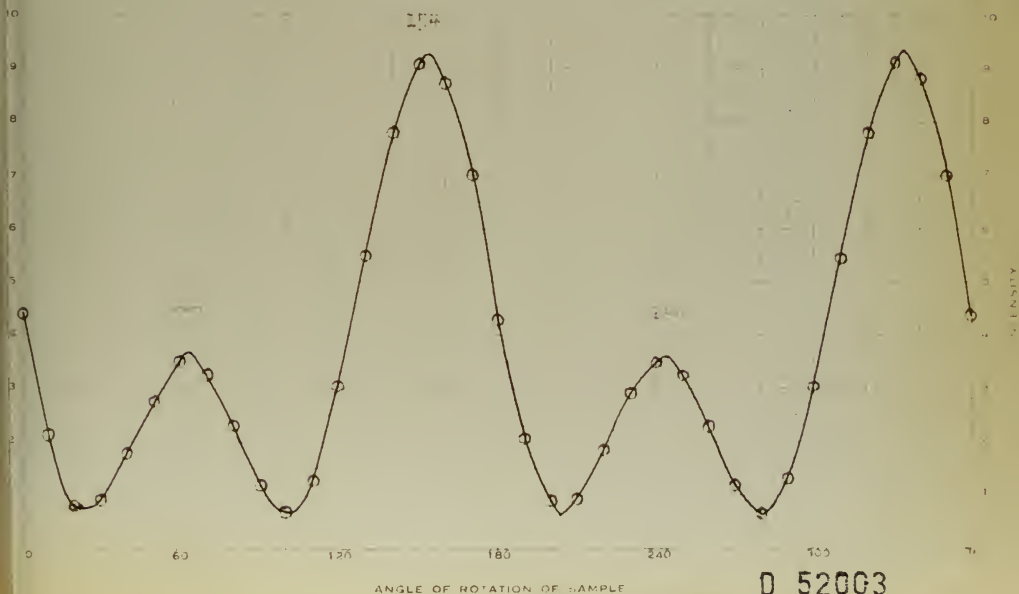
M <sub>2</sub>	45.30	R <sub>2</sub>	45.75
M <sub>3</sub>	45.30	R <sub>3</sub>	45.65
ΔM	13.30	ΔR	- .05

AV ΔM 13.25 PHASE SHIFT

AV ΔR - .10 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







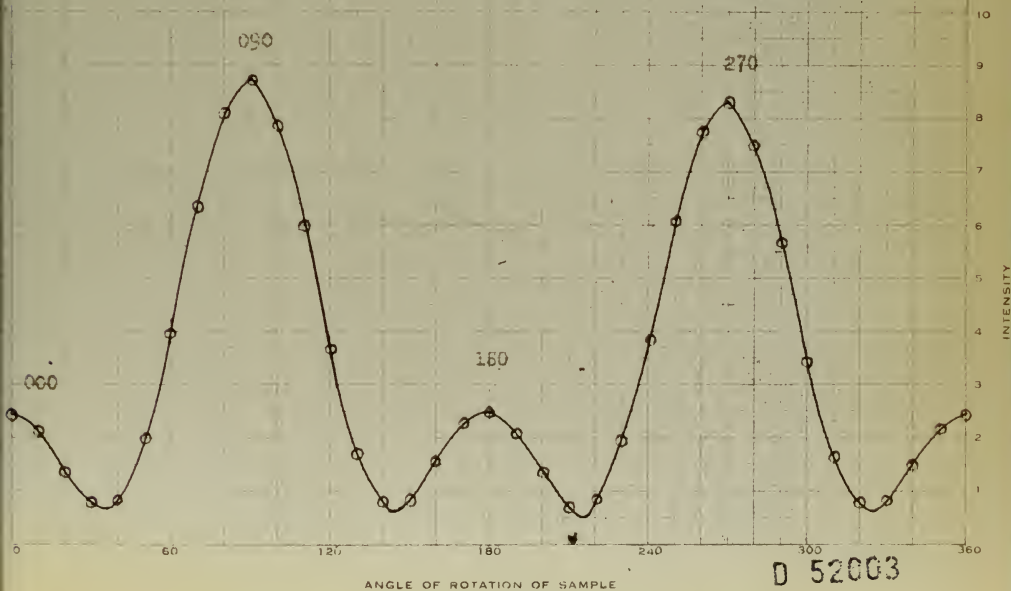
SAMPLE NO. 1A		PPM 2		DATE 2 JAN 1960			
CRYSTAL NO. 1		SURFACE FILM CONDITION					
@ 090		@ 180		@ 270		@ 360	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.1	45.7	37.3	45.3	50.8	45.3	37.1	45.7
51.1	45.7	37.9	45.3	51.0	45.6	37.3	45.7
51.10	45.70	37.35	45.30	50.90	45.45	37.20	45.70

M <sub>1</sub>	51.10	R <sub>1</sub>	45.70
M <sub>2</sub>	37.95	R <sub>2</sub>	45.80
$\Delta M_2$	13.25	$\Delta R_2$	-.10

M <sub>3</sub>	50.90	R <sub>3</sub>	45.45
M <sub>4</sub>	37.20	R <sub>4</sub>	45.70
$\Delta M_{34}$	13.70	$\Delta R_{34}$	-.25

AV  $\Delta M$  13.48 PHASE SHIFT  
 AV  $\Delta R$  -.18 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



@ 335		@ 065		@ 155		@ 245	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.2	45.2	35.3	45.7	49.8	45.6	35.7	46.0
50.0	45.7	36.1	45.5	49.6	45.5	36.0	46.0
AV 50.10	45.45	36.00	45.60	49.70	45.55	35.85	46.00

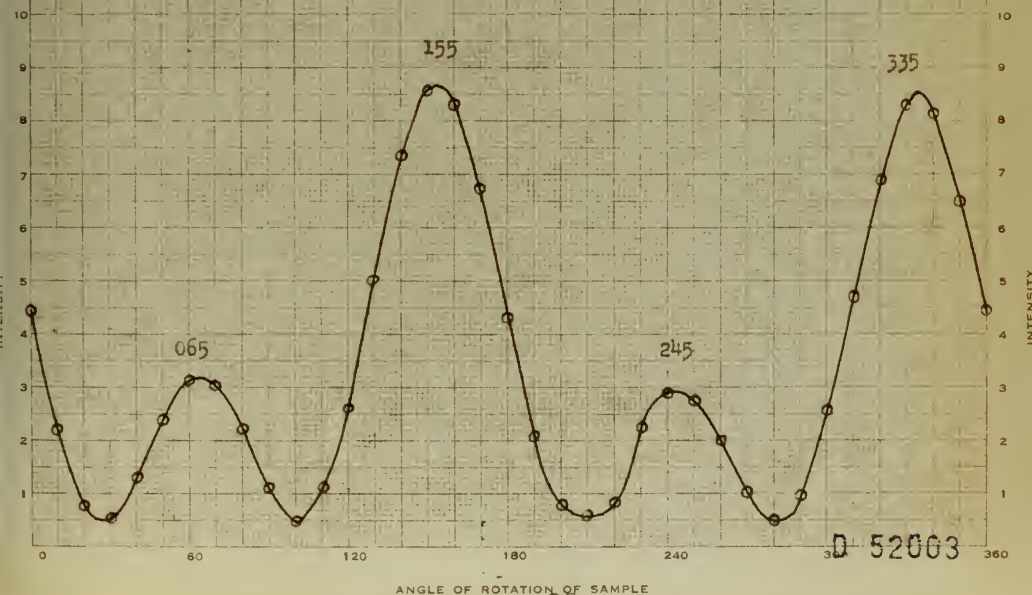
M <sub>1</sub>	50.10	R <sub>1</sub>	45.45
M <sub>2</sub>	36.00	R <sub>2</sub>	45.60
$\Delta M_{12}$	14.10	$\Delta R_{12}$	-.15

M <sub>3</sub>	49.70	R <sub>3</sub>	45.55
M <sub>4</sub>	35.85	R <sub>4</sub>	46.00
$\Delta M_{34}$	13.85	$\Delta R_{34}$	-.45

AV  $\Delta M$  13.98 PHASE SHIFT  
AV  $\Delta R$  -.30 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 1A

6 PPM  $N_2$ 

DATE 29 MARCH 1952 PM

9th AUTOCLAVE OF 15 MIN. @ 233°C

CRYSTAL NO 1

SURFACE FILM CONDITION

@ 299		@ 029		@ 119		@ 209	
M1	R1	M2	R2	M3	R3	M4	R4
51.3	44.8	36.6	45.5	50.6	45.3	36.3	45.6
51.6	45.3	37.0	45.5	50.6	45.1	36.6	45.4
51.45	45.05	36.80	45.55	50.60	45.20	36.45	45.50

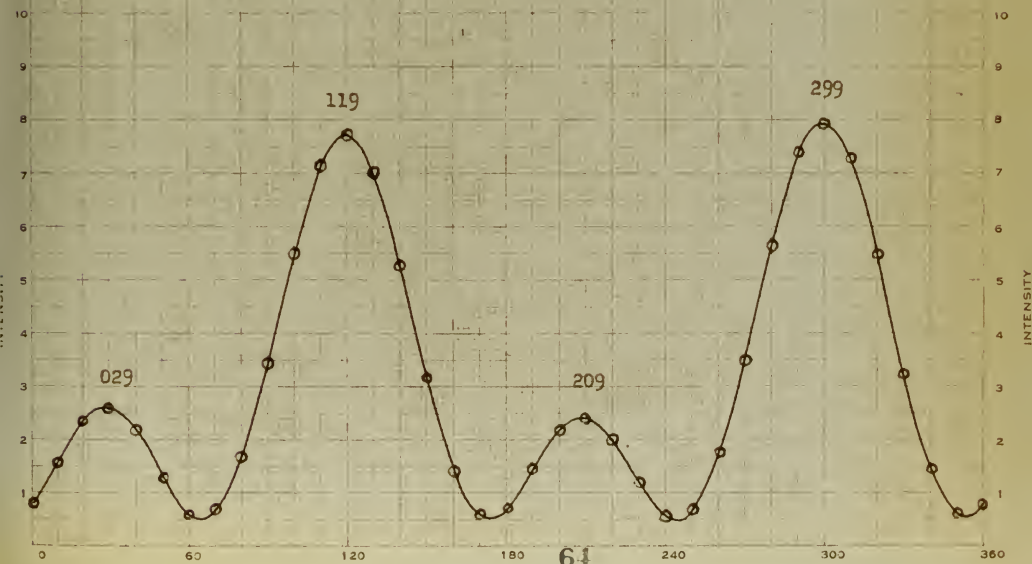
M <sub>1</sub> 51.45	R <sub>1</sub> 45.05
M <sub>2</sub> 36.80	R <sub>2</sub> 45.55
$\Delta M_{12}$ 14.65	$\Delta R_{12}$ - .50

M <sub>3</sub> 50.60	R <sub>3</sub> 45.20
M <sub>4</sub> 36.45	R <sub>4</sub> 45.50
$\Delta M_{34}$ 14.15	$\Delta R_{34}$ - .30

AV  $\Delta M$  14.40 PHASE SHIFTAV  $\Delta R$  - .40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





CRYSTAL NO. 1 SURFACE FILM CONDITION 10th AUTOCLAVE OF 15 MIN. @ 233°C

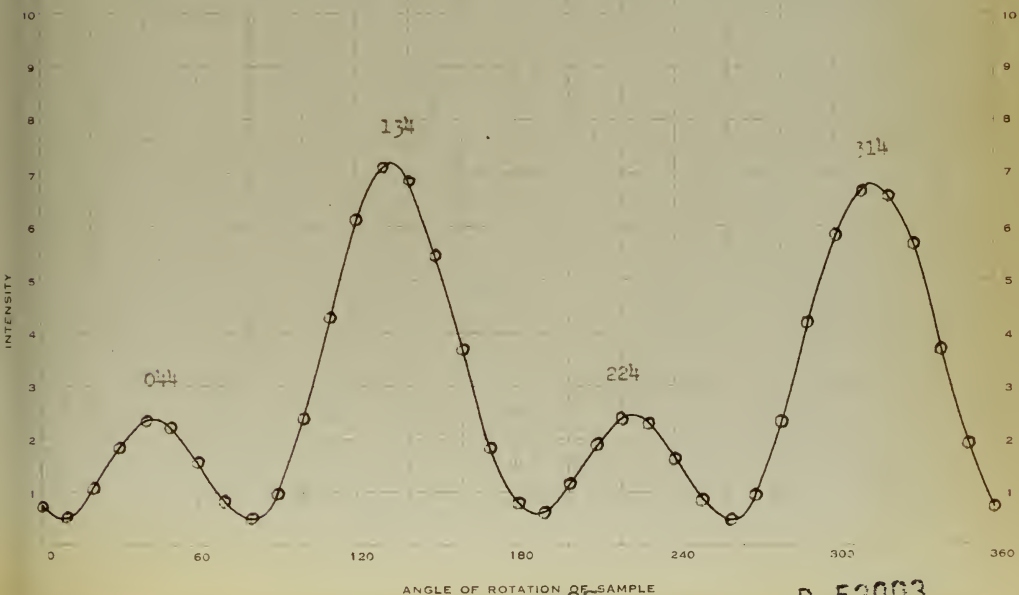
@ 314		@ 044		@ 134		@ 224	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.9	45.2	36.8	45.8	50.2	45.5	36.0	45.8
50.7	45.5	37.0	45.7	50.6	45.8	36.4	45.9
Av 50.80	45.35	36.90	45.75	50.40	45.65	36.20	45.85

M <sub>1</sub> 50.80	R <sub>1</sub> 45.35
M <sub>2</sub> 36.90	R <sub>2</sub> 45.75
ΔM <sub>2</sub> 13.90	ΔR <sub>2</sub> -.40

M <sub>1</sub> 50.40	R <sub>1</sub> 45.65
M <sub>2</sub> 36.20	R <sub>2</sub> 45.85
ΔM <sub>2</sub> 14.20	ΔR <sub>2</sub> -.20

Av ΔM 14.05 PHASE SHIFT  
 Av ΔR -.30 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 1A

6 PPM  $N_2$ 

DATE 1 APR 1952 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

11th AUTOCLAVE OF 15 MIN. @ 233°C

@ 325		@ 055		@ 145		@ 235	
M1	R	M2	R2	M3	R3	M4	R4
48.8	45.5	35.3	45.4	48.5	45.5	34.7	45.4
48.6	45.4	35.3	45.6	48.6	45.5	34.8	45.5
48.70	45.45	35.30	45.50	48.55	45.50	34.75	45.45

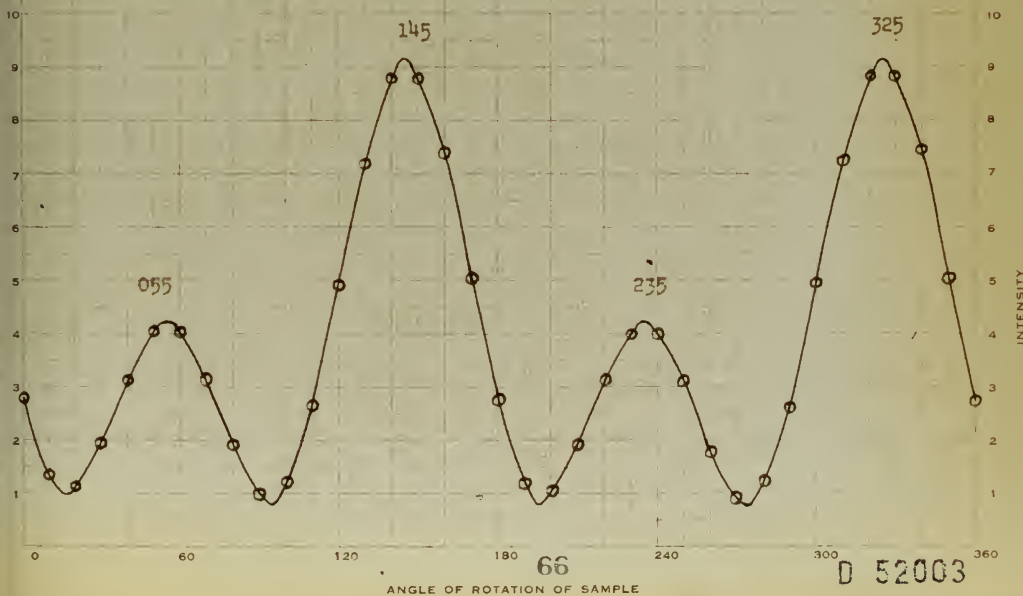
M1	48.70	R1	45.45
M2	35.30	R2	45.50
$\Delta M_{12}$	13.40	$\Delta R_{12}$	-.05

M3	48.55	R3	45.50
M4	34.75	R4	45.45
$\Delta M_{34}$	13.80	$\Delta R_{34}$	+.05

AV  $\Delta M$  13.60 PHASE SHIFTAV  $\Delta R$  .00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO

PPM

DATE

CRYSTAL NO

SURFACE FILM CONDITION

AV

@ 11°		@ 11°		@ 20°		@ 20°	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
40.8	45.6	35.5	45.5	49.5	45.5	35.1	44.4
49.4	45.5	35.8	45.5	49.0	45.5	35.5	44.5
49.60	45.55	35.35	45.55	49.25	45.50	35.35	44.45

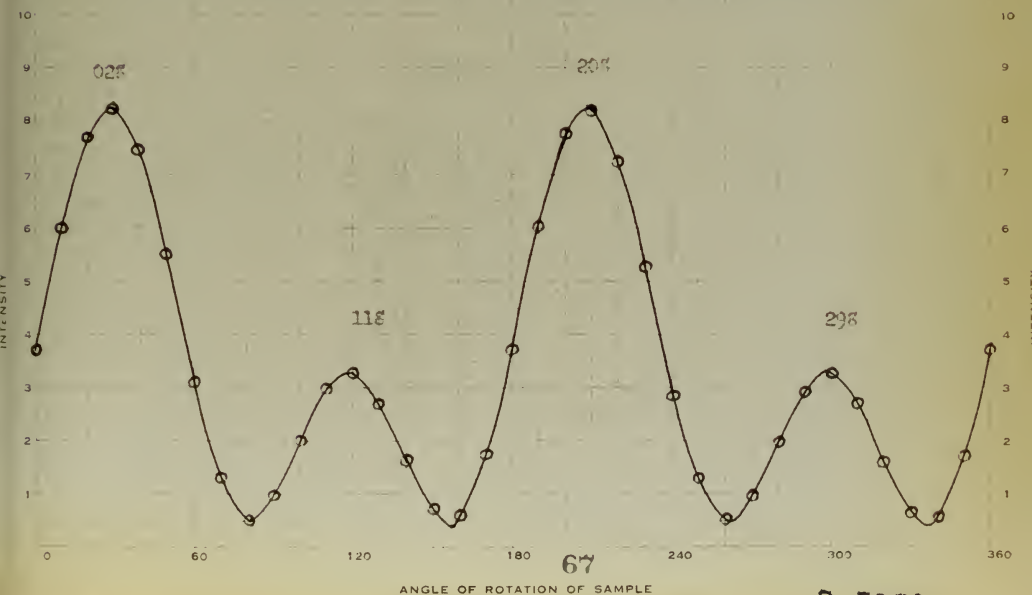
M <sub>1</sub>	49.60	R <sub>1</sub>	45.55
M <sub>2</sub>	35.35	R <sub>2</sub>	45.55
$\Delta M_{12}$	13.25	$\Delta R_{12}$	.00

M <sub>3</sub>	49.25	R <sub>3</sub>	45.50
M <sub>4</sub>	35.35	R <sub>4</sub>	44.45
$\Delta M_{34}$	13.90	$\Delta R_{34}$	.05

AV  $\Delta M$  13.93 PHASE SHIFTAV  $\Delta R$  .03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE No. 1A

6 PPM  $N_2$ 

DATE 3 APRIL 1952 PM

CRYSTAL No. 1

SURFACE FILM CONDITION

13th AUTOCLAVE OF 15 MIN. @ 233°C

@ 328		@ 058		@ 148		@ 238	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.8	45.7	35.3	46.0	49.0	45.5	35.0	45.7
49.2	45.6	35.3	45.6	48.9	45.7	34.8	45.8
AV. 49.00	45.65	35.30	45.80	48.95	45.60	34.90	45.75

M <sub>1</sub>	49.00	R <sub>1</sub>	45.65
M <sub>2</sub>	35.30	R <sub>2</sub>	45.80
$\Delta M_{12}$	13.70	$\Delta R_{12}$	- .15

M <sub>3</sub>	48.95	R <sub>3</sub>	45.60
M <sub>4</sub>	34.90	R <sub>4</sub>	45.75
$\Delta M_{34}$	14.05	$\Delta R_{34}$	- .15

AV  $\Delta M$  13.88

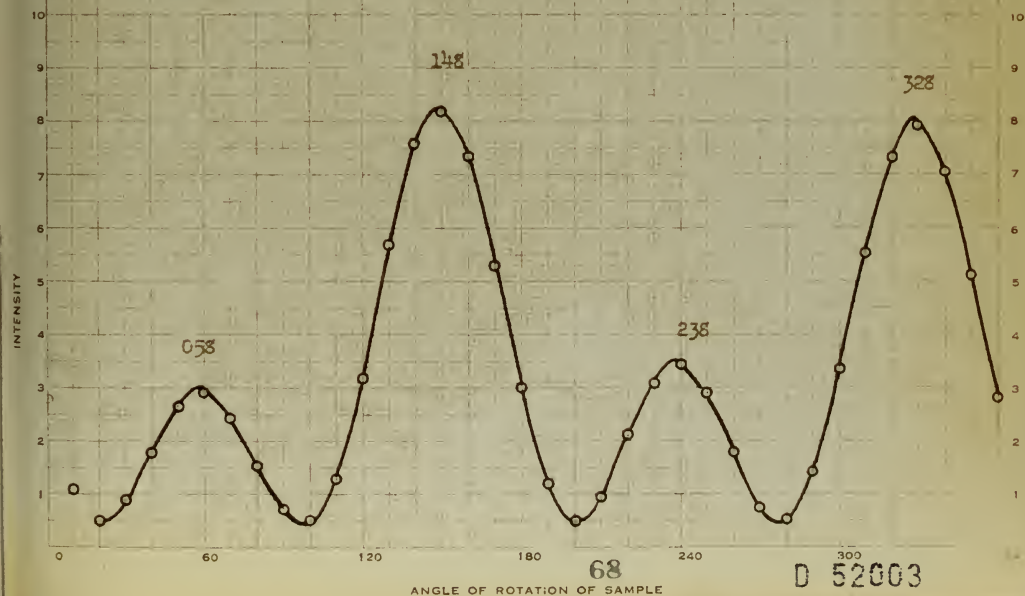
PHASE SHIFT

AV  $\Delta R$  -.15

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





SAMPLE NO 11

6 PPM<sup>1/2</sup>

DATE 5 APRIL 1952 EW

CRYSTAL NO 1

SURFACE FILM CONDITION

15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 150		@ 240		@ 330		@ 060	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.1	45.6	36.8	45.8	50.2	45.6	36.6	45.8
51.1	45.6	36.7	45.7	50.6	45.7	36.6	45.9
AV. 51.10	45.60	36.75	45.75	50.40	45.65	36.60	45.85

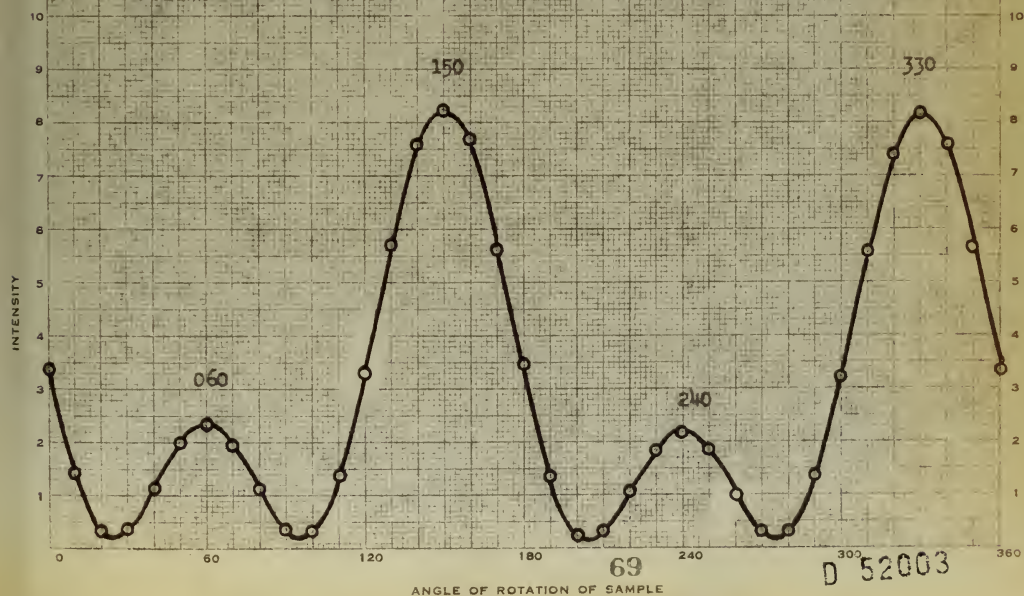
M <sub>1</sub>	51.10	R <sub>1</sub>	45.60
M <sub>2</sub>	36.75	R <sub>2</sub>	45.75
$\Delta M_{12}$	14.35	$\Delta R_{12}$	-.15

M <sub>3</sub>	50.40	R <sub>3</sub>	45.65
M <sub>4</sub>	36.60	R <sub>4</sub>	45.85
$\Delta M_{34}$	13.80	$\Delta R_{34}$	-.20

AV  $\Delta M$  14.08 PHASE SHIFTAV  $\Delta R$  -.18 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

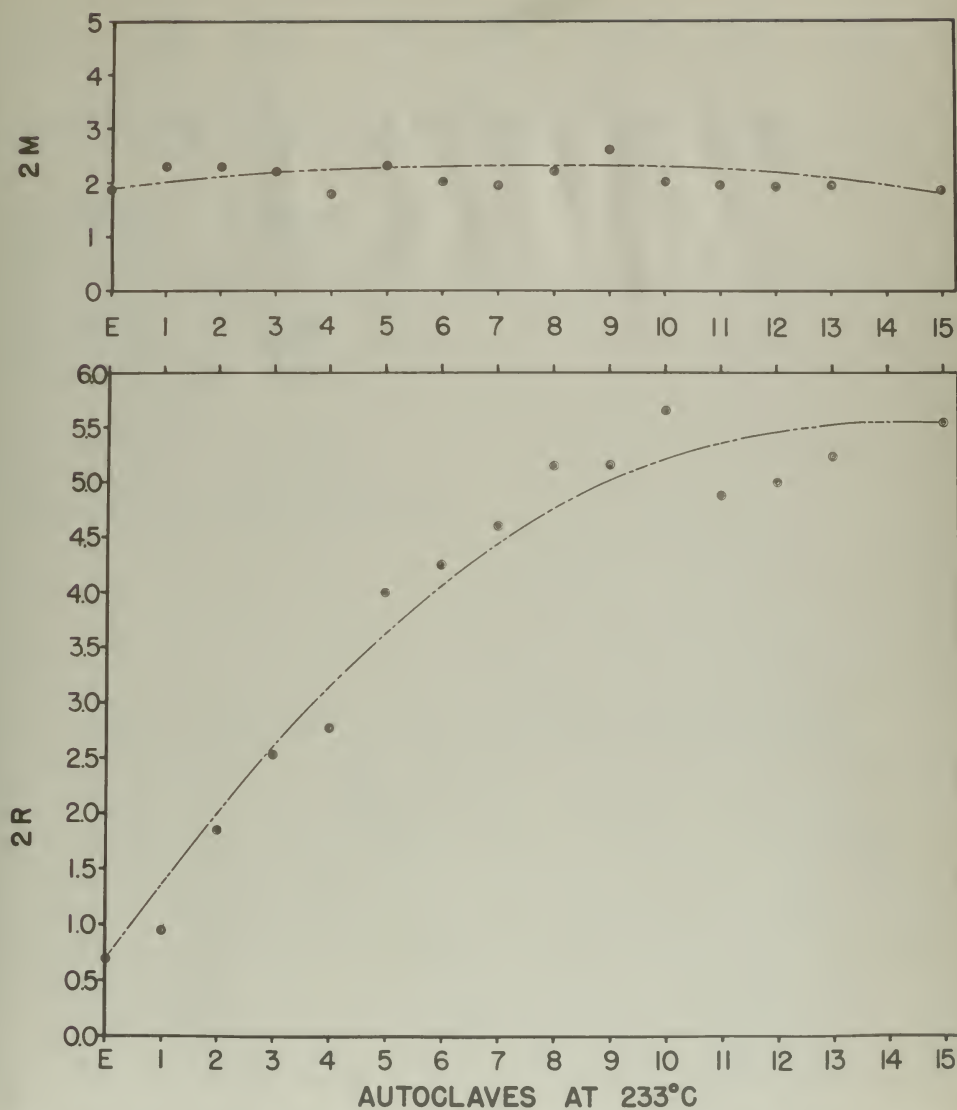
GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003







PHASE SHIFT ( $2m$ ) AND ROTATION OF PLANE OF  
POLARIZATION ( $2r$ ) VS. CORROSION TIME  
FOR SAMPLE NO. 1A-2



SAMPLE NO 1A

6 PPM N<sub>2</sub>

DATE 6 March 1952 PM

CRYSTAL NO 2

SURFACE FILM CONDITION AS ELECTRO-POLISHED

@ 324		@ 052		@ 144		@ 232	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
43.5	45.9	42.0	45.0	43.7	45.8	41.6	45.3
43.6	45.6	41.6	45.1	43.6	46.0	41.7	45.1
AV 43.55	45.75	41.80	45.05	43.65	45.90	41.65	45.20

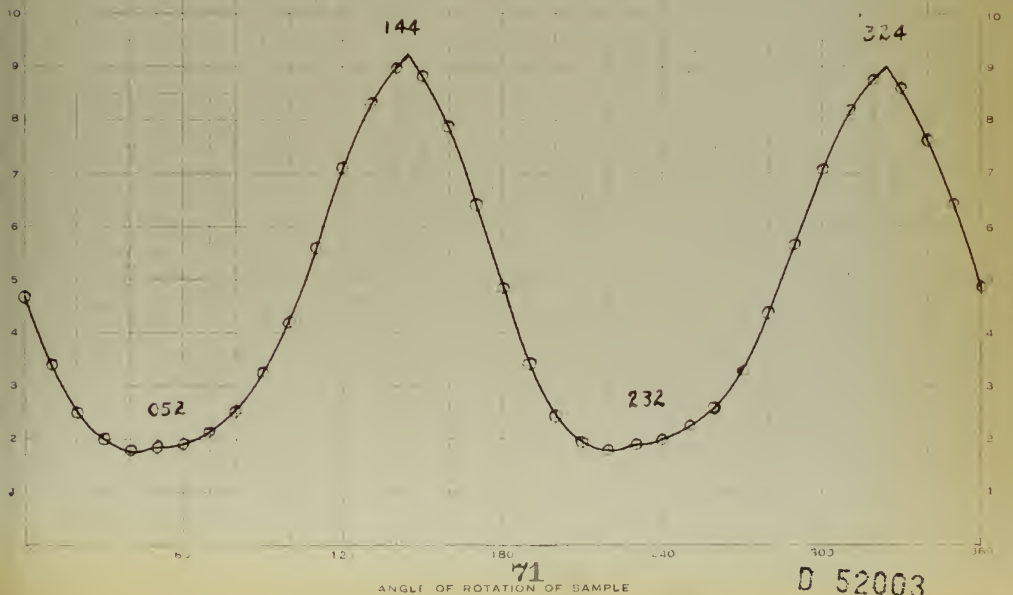
M <sub>1</sub>	43.55	R <sub>1</sub>	45.75
M <sub>2</sub>	41.80	R <sub>2</sub>	45.05
$\Delta M_{12}$	1.75	$\Delta R_{12}$	.70

M <sub>3</sub>	43.65	R <sub>3</sub>	45.90
M <sub>4</sub>	41.65	R <sub>4</sub>	45.20
$\Delta M_{34}$	2.00	$\Delta R_{34}$	0.70

AV  $\Delta M$  1.88 PHASE SHIFTAV  $\Delta R$  .70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO. 1A 6 PPM  $N_2$  DATE 22 March 1952  
 CRYSTAL NO. 2 SURFACE FILM CONDITION 1st AUTOCLAVE 15 Min. at 233°C

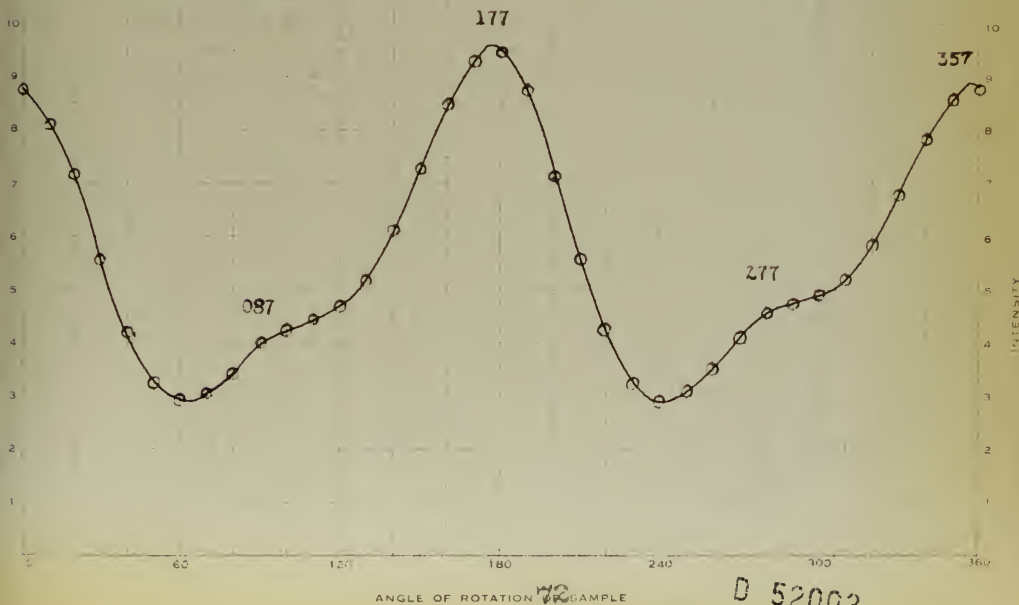
357		087		177		277	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
43.2	45.8	41.2	45.4	44.0	46.8	41.0	45.2
43.3	46.3	41.0	45.5	43.3	46.7	41.4	45.7
AV 43.25	46.05	41.10	45.45	43.65	46.75	41.20	45.45

M<sub>1</sub> 43.45 R<sub>1</sub> 46.05  
 M<sub>2</sub> 41.10 R<sub>2</sub> 45.45  
 $\Delta M$  2.15  $\Delta R$  0.60

M<sub>3</sub> 43.55 R<sub>3</sub> 46.75  
 M<sub>4</sub> 41.20 R<sub>4</sub> 45.45  
 $\Delta M$  2.45  $\Delta R$  1.30

AV  $\Delta M$  2.30 PHASE SHIFT  
 AV  $\Delta R$  0.95 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



CRYSTAL NO 2

SURFACE FILM CONDITION

Av

@ 0°		@ 110°		@ 210°		@ 270°	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
40.3	40.7	41.7	44.8	43.7	45.5	41.4	44.5
44.0	45.6	41.4	44.7	43.5	44.4	40.7	44.0
43.00	40.65	41.20	44.60	43.30	44.55	41.00	44.70

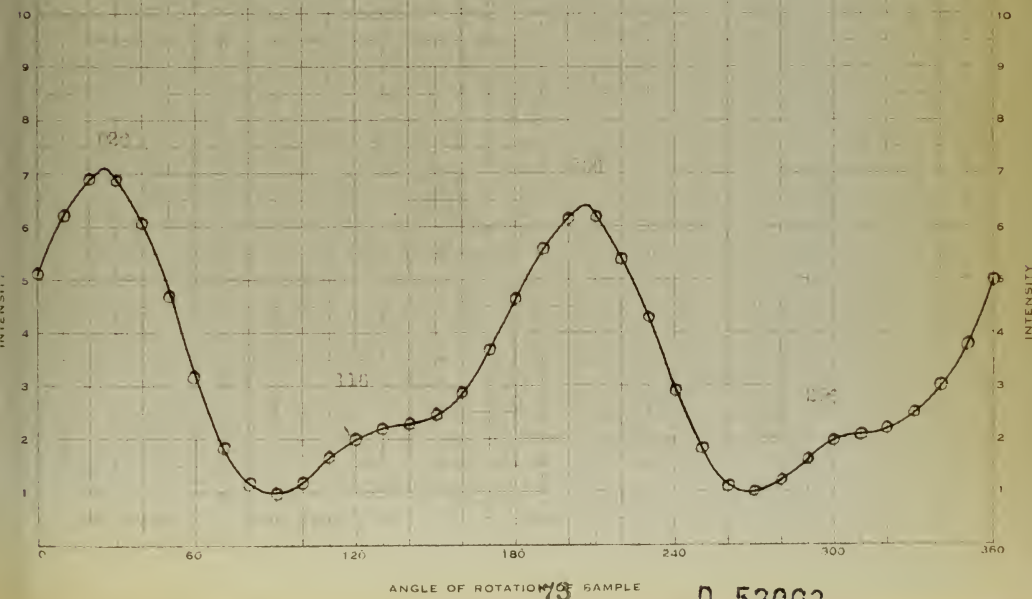
M <sub>1</sub>	43.40	R <sub>1</sub>	40.55
M <sub>2</sub>	41.20	R <sub>2</sub>	44.60
$\Delta M_{12}$	2.20	$\Delta R_{12}$	4.15

M <sub>3</sub>	43.30	R <sub>3</sub>	44.55
M <sub>4</sub>	41.00	R <sub>4</sub>	44.70
$\Delta M_{34}$	2.30	$\Delta R_{34}$	1.15

Av  $\Delta M$  2.30 PHASE SHIFTAv  $\Delta R$  1.85 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





CRYSTAL NO

SURFACE FILM CONDITION

AV

@ 317		@ 317		@ 317		@ 317	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.5	46.7	42.3	44.1	44.1	46.8	42.2	44.1
44.3	46.6	42.2	44.0	44.0	46.6	42.7	44.4
44.40	46.65	42.25	44.05	44.20	46.70	42.45	44.25

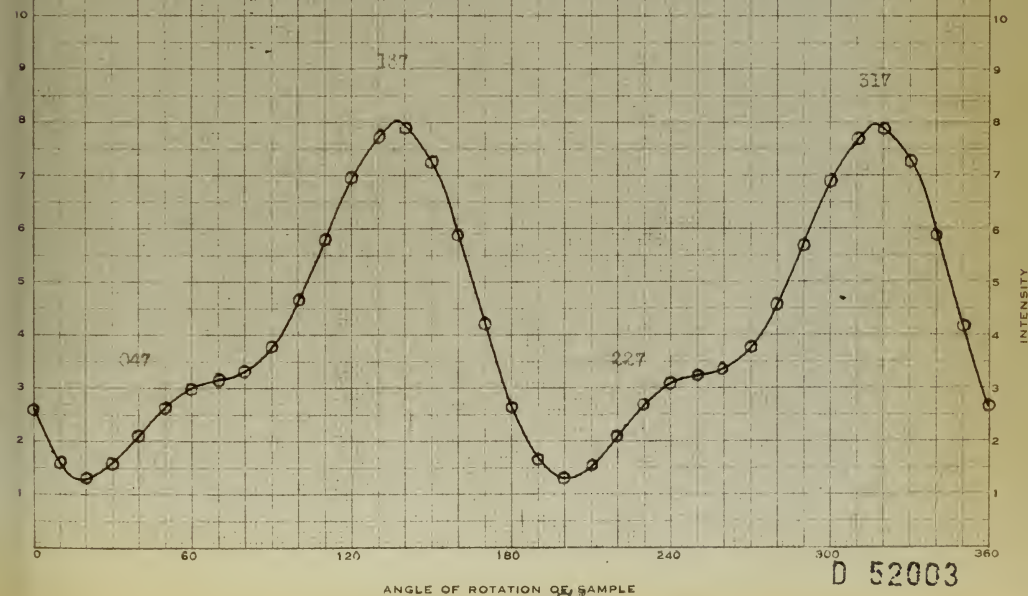
M <sub>1</sub>	44.40	R <sub>1</sub>	46.65
M <sub>2</sub>	42.25	R <sub>2</sub>	44.05
$\Delta M_{12}$	2.15	$\Delta R_{12}$	2.60

M <sub>3</sub>	44.20	R <sub>3</sub>	46.70
M <sub>4</sub>	42.45	R <sub>4</sub>	44.25
$\Delta M_{34}$	2.25	$\Delta R_{34}$	2.45

AV  $\Delta M$  2.20 PHASE SHIFTAV  $\Delta R$  2.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE



CRYSTAL NO. 2 SURFACE FILM CONDITION 4th AUTOCLAVE OF 15 MIN. at 233°

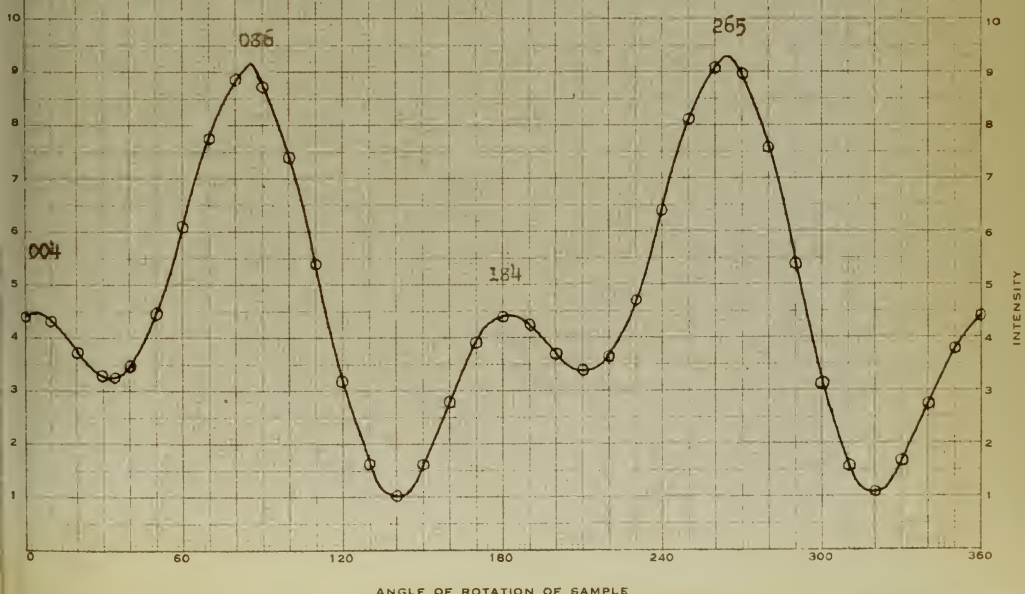
@ 265		@ 004		@ 026		@ 184	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
42.8	47.0	41.1	43.9	42.2	46.7	40.5	44.2
42.8	47.0	40.8	44.1	42.4	46.7	40.7	44.1
42.80	47.00	40.95	44.00	42.30	46.70	40.60	44.15

M <sub>1</sub>	42.80	R <sub>1</sub>	47.00
M <sub>2</sub>	40.95	R <sub>2</sub>	44.00
ΔM <sub>12</sub>	1.85	ΔR <sub>12</sub>	3.00

M <sub>3</sub>	42.30	R <sub>3</sub>	46.70
M <sub>4</sub>	40.60	R <sub>4</sub>	44.15
ΔM <sub>34</sub>	1.70	ΔR <sub>34</sub>	2.55

AV ΔM 1.78 PHASE SHIFT  
 AV ΔR 2.78 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





@ 238		@ 334		@ 053		@ 153	
M	R	M <sub>1</sub>	R	M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>
43.2	47.4	40.0	43.5	42.6	47.7	40.1	43.8
43.0	47.4	40.6	43.4	42.5	47.6	40.4	43.4
AV 43.10	47.40	40.75	43.45	42.70	47.65	40.45	43.60

M	43.10	R	47.40
M	40.75	R	43.45
$\Delta M$	2.35	$\Delta R$	3.95

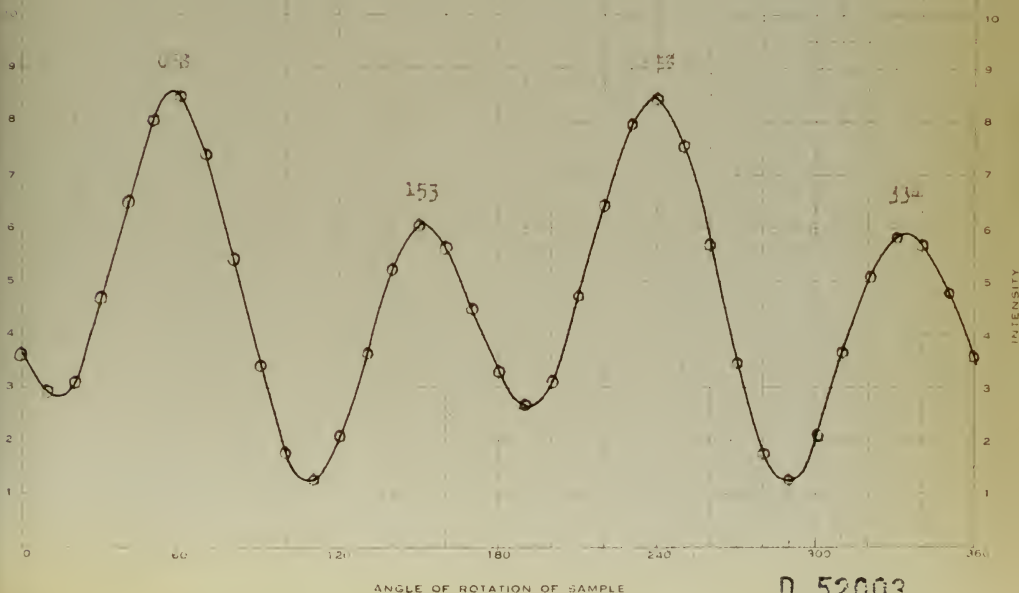
M <sub>1</sub>	42.70	R <sub>1</sub>	47.65
M <sub>2</sub>	40.45	R <sub>1</sub>	43.60
$\Delta M$	2.25	$\Delta R$	4.05

AV  $\Delta M$  2.30 PHASE SHIFT

AV  $\Delta R$  4.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





SAMPLE NO. 1

PPM

DATE

CRYSTAL NO.

SURFACE FILM CONDITION

225

(a) 225		(a) 225		(a) 225		(a) 225	
M	R	M	R	M	R	M	R
44.2	47.8	42.1	43.7	44.0	47.8	42.0	43.6
44.3	47.8	42.1	43.5	43.9	47.8	42.0	43.5
AV 44.25	47.8	42.1	43.60	43.95	47.80	42.00	43.50

M	44.25	R	47.80
M	42.10	R	43.60
$\Delta M$	2.15	$\Delta R$	4.20

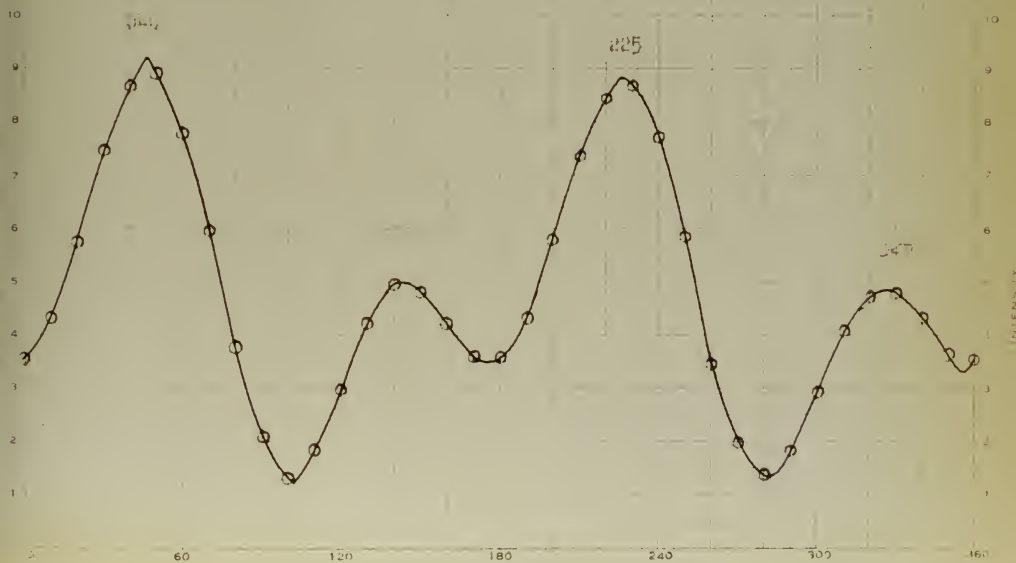
M	44.25	R	47.80
M	42.10	R	43.60
$\Delta M_{24}$	1.85	$\Delta R$	4.20

AV  $\Delta M$  2.00 PHASE SHIFT

AV  $\Delta R$  4.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003



CRYSTAL NO. 2

SURFACE FILM CONDITION

7th AUTOCLAVE OF 15 MIN. @ 233°C

@ 341		@ 083		@ 161		@ 261	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.8	47.7	43.0	43.0	44.4	47.6	42.3	43.1
44.7	47.7	42.7	43.2	44.2	47.6	42.4	42.9
44.75	47.70	42.85	43.10	44.30	47.60	42.35	43.00

M <sub>1</sub>	44.75	R <sub>1</sub>	47.70
M <sub>2</sub>	42.95	R <sub>2</sub>	43.10
$\Delta M_2$	1.90	$\Delta R_2$	4.60

M <sub>3</sub>	44.30	R <sub>3</sub>	47.60
M <sub>4</sub>	42.35	R <sub>4</sub>	43.00
$\Delta M_{34}$	1.95	$\Delta R_{31}$	4.60

AV  $\Delta M$  1.93

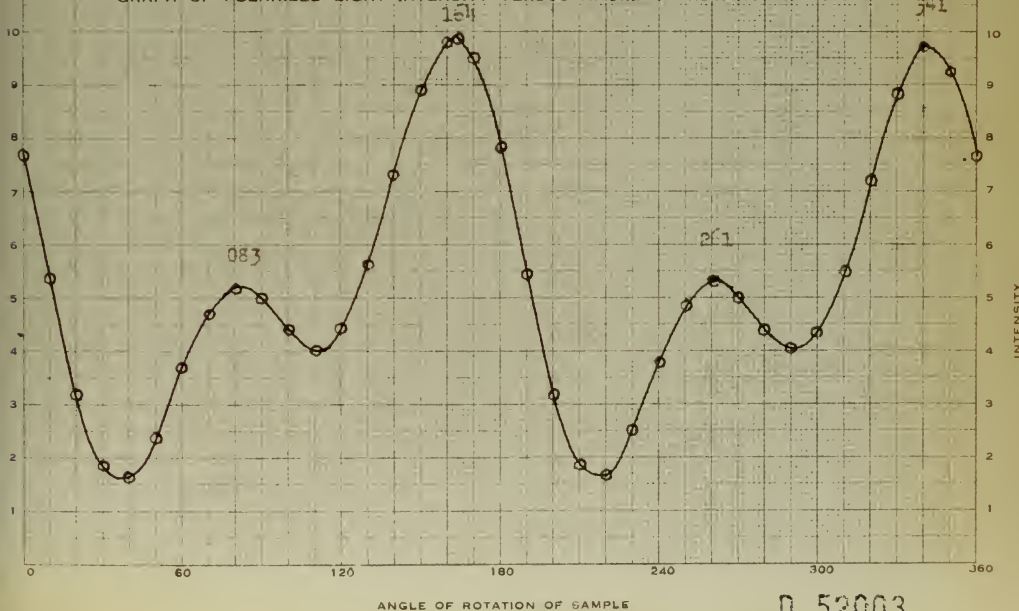
PHASE SHIFT

AV  $\Delta R$  4.60

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



CRYSTAL NO 2

SURFACE FILM CONDITION

8th AUTOCLAVE OF 15 MIN. @ 233°C

@ 047		@ 145		@ 227		@ 325	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.7	48.2	42.0	43.4	44.3	48.4	42.3	42.9
44.6	48.3	42.4	43.2	44.3	48.2	42.4	43.0
44.65	48.25	42.20	43.30	44.30	48.30	42.35	42.95

AV

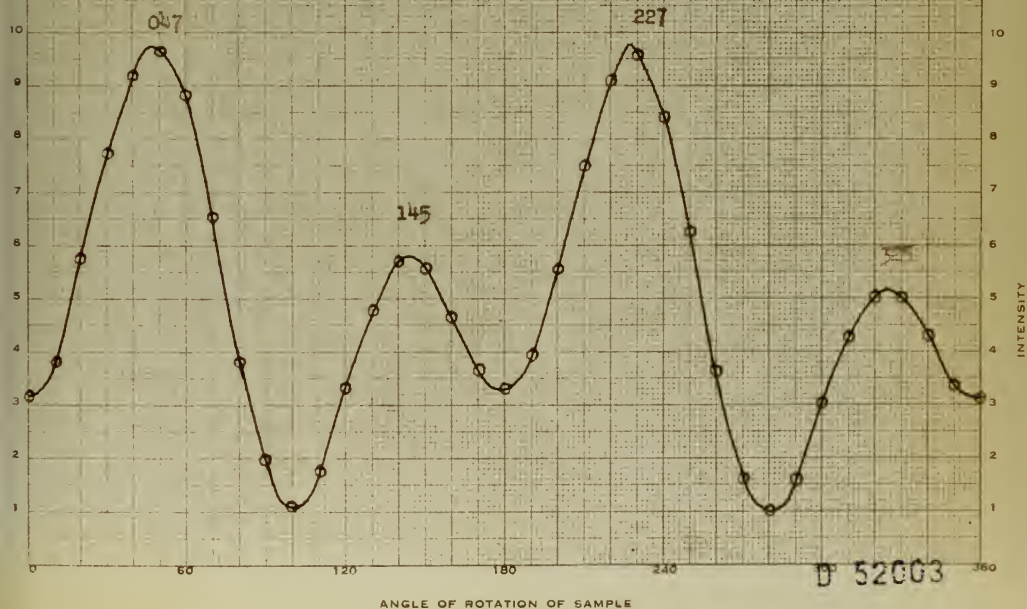
M <sub>1</sub>	44.65	R <sub>1</sub>	48.25
M <sub>2</sub>	42.20	R <sub>2</sub>	43.30
$\Delta M_{12}$	2.45	$\Delta R_{12}$	4.95

M <sub>3</sub>	44.30	R <sub>3</sub>	48.30
M <sub>4</sub>	42.35	R <sub>4</sub>	42.95
$\Delta M_{34}$	1.95	$\Delta R_{34}$	5.35

AV  $\Delta M$  2.20 PHASE SHIFTAV  $\Delta R$  5.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE





CRYSTAL No 2

SURFACE FILM CONDITION

10% ANGLE OF 15 MIN. 4 33°C

@ 011		@ 110		@ 112		@ 289	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.3	48.3	42.5	43.0	45.2	48.1	42.5	42.9
45.5	48.2	42.5	43.1	44.7	48.0	42.8	43.0
45.40	48.25	42.50	43.05	44.95	48.05	42.65	42.95

M <sub>1</sub>	45.40	R <sub>1</sub>	48.25
M <sub>2</sub>	42.50	R <sub>2</sub>	43.05
$\Delta M_{12}$	2.90	$\Delta R_{12}$	5.20

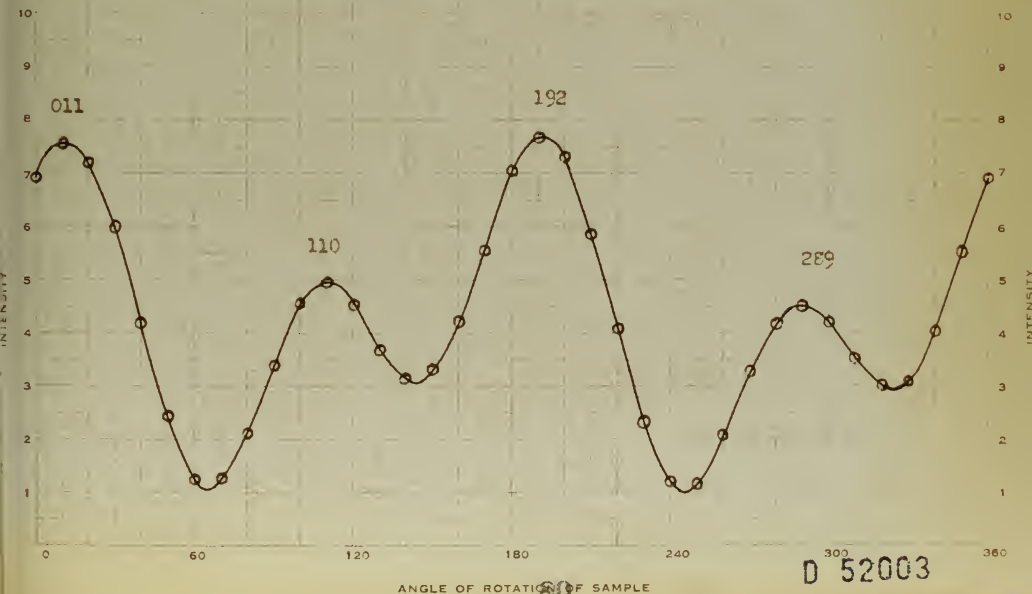
M <sub>3</sub>	44.95	R <sub>3</sub>	48.05
M <sub>4</sub>	42.65	R <sub>4</sub>	42.95
$\Delta M_{34}$	2.30	$\Delta R_{34}$	5.10

AV  $\Delta M$  2.60 PHASE SHIFT

AV  $\Delta R$  5.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL NO. 2 SURFACE FILM CONDITION 10% AUTO SLAVE OF 15 MIN. @ 233 C

@ 029		@ 122		@ 216		@ 303	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.8	48.0	42.9	42.6	44.0	48.4	41.9	42.6
45.0	48.4	42.3	42.8	43.6	48.3	42.3	42.5
44.90	48.20	42.50	42.70	43.80	48.35	42.10	42.55

M <sub>1</sub>	44.90	R <sub>1</sub>	48.20
M <sub>2</sub>	42.50	R <sub>2</sub>	42.70
$\Delta M_{12}$	2.40	$\Delta R_{12}$	5.50

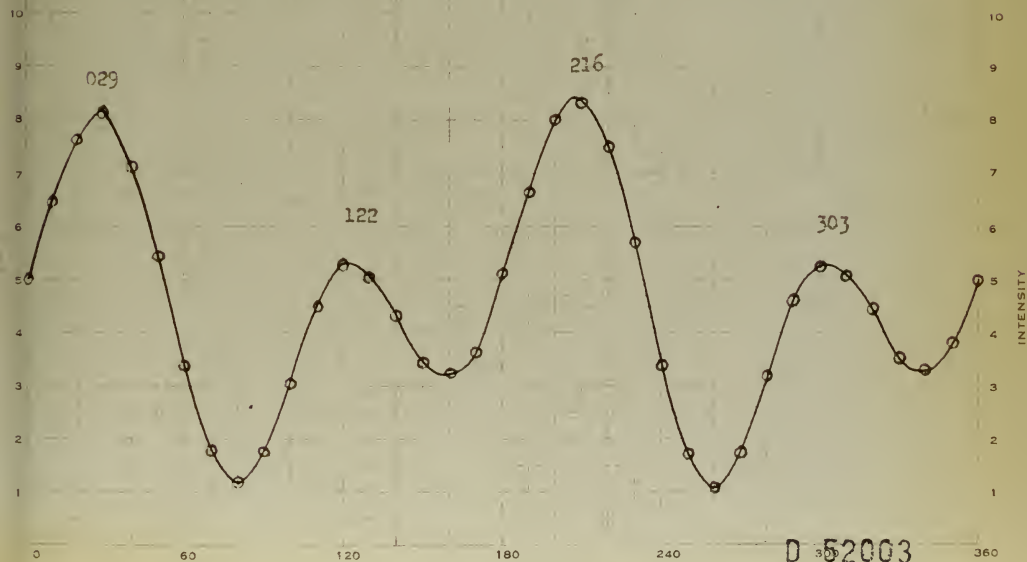
M <sub>3</sub>	43.80	R <sub>3</sub>	48.35
M <sub>4</sub>	42.10	R <sub>4</sub>	42.55
$\Delta M_{34}$	1.70	$\Delta R_{34}$	5.80

AV  $\Delta M$  2.00 PHASE SHIFT

AV  $\Delta R$  5.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE NO 1A

6 PPM N<sub>2</sub>

DATE 1 APR 11 1950 PM

CRYSTAL NO 2

SURFACE FILM CONDITION

11th AUTOCLAVE OF 1- MIN. @ 233°C

@ 042		@ 137		@ 222		@ 316	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>1</sub>	M <sub>4</sub>	R <sub>4</sub>
43.0	47.8	40.9	42.8	42.7	47.9	40.7	43.0
43.2	47.3	41.2	43.0	42.3	47.8	40.7	43.0
43.10	47.80	41.05	42.90	42.50	47.85	40.70	43.00

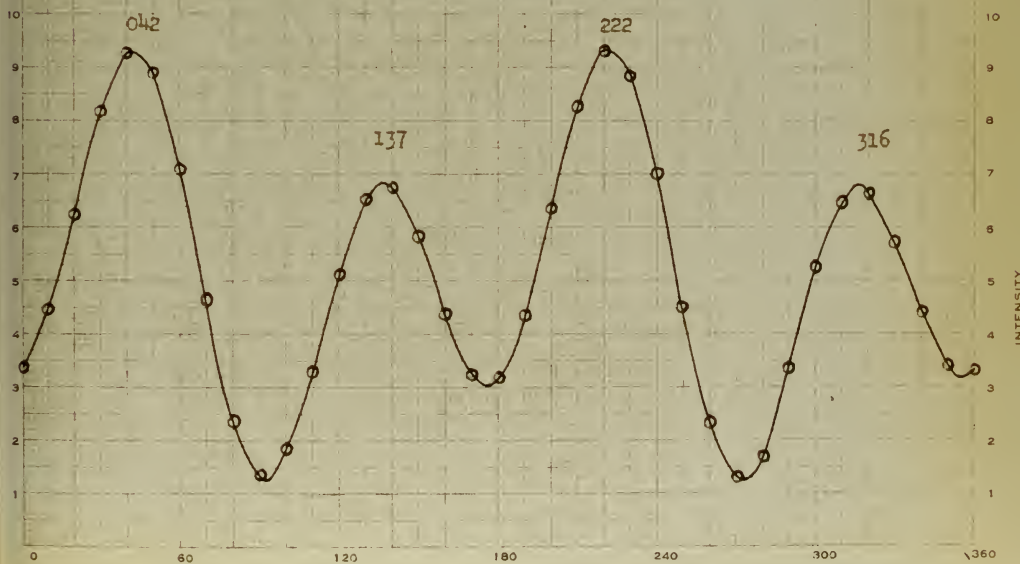
M <sub>1</sub>	43.10	R <sub>1</sub>	47.80
M <sub>2</sub>	41.05	R <sub>2</sub>	42.90
$\Delta M_{12}$	2.05	$\Delta R_{12}$	4.90

M <sub>3</sub>	42.50	R <sub>3</sub>	47.85
M <sub>4</sub>	40.70	R <sub>4</sub>	43.00
$\Delta M_{34}$	1.80	$\Delta R_{34}$	4.85

AV  $\Delta M$  1.93 PHASE SHIFTAV  $\Delta R$  4.88 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



SAMPLE No 1A

PPM

DATE

CRYSTAL No

SURFACE FILM CONDITION

1250-17-01A7 OF 19 A.T. 45372

@ 283		@ 618		@ 102		@ 198	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
43.4	48.0	41.5	43.0	42.3	47.8	41.1	45.0
43.3	47.9	41.4	42.9	43.1	48.2	41.0	43.0
43.35	47.95	41.45	42.95	42.95	48.00	41.05	45.00

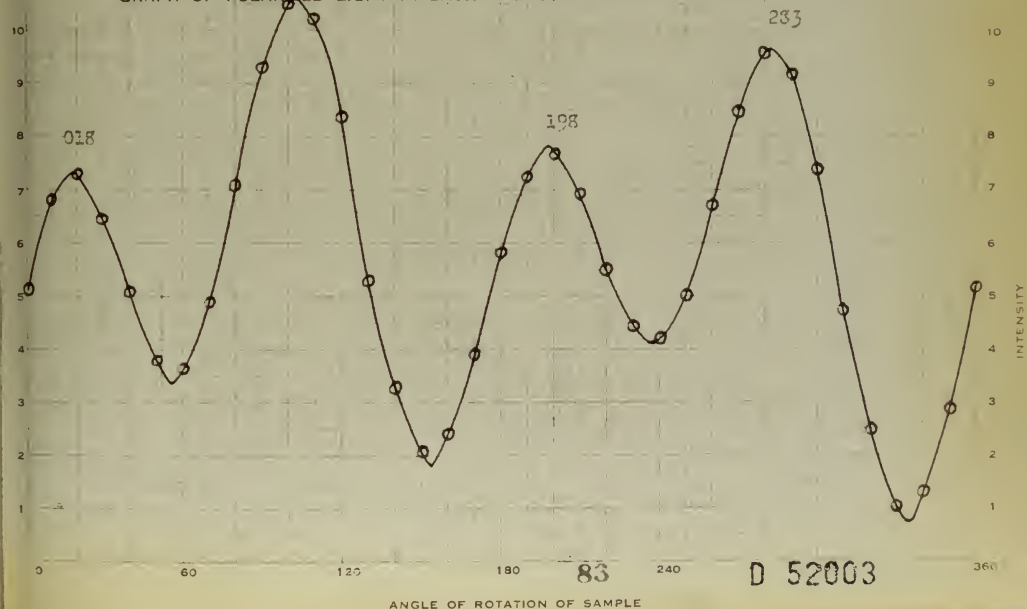
M <sub>1</sub>	43.35	R <sub>1</sub>	47.95
M <sub>2</sub>	41.45	R <sub>2</sub>	42.95
$\Delta M_{12}$	1.90	$\Delta R_{12}$	5.00

M <sub>3</sub>	42.95	R <sub>3</sub>	48.00
M <sub>4</sub>	41.05	R <sub>4</sub>	43.00
$\Delta M_{34}$	1.90	$\Delta R_{34}$	5.00

AV  $\Delta M$  1.90 PHASE SHIFTAV  $\Delta R$  5.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

102  
GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







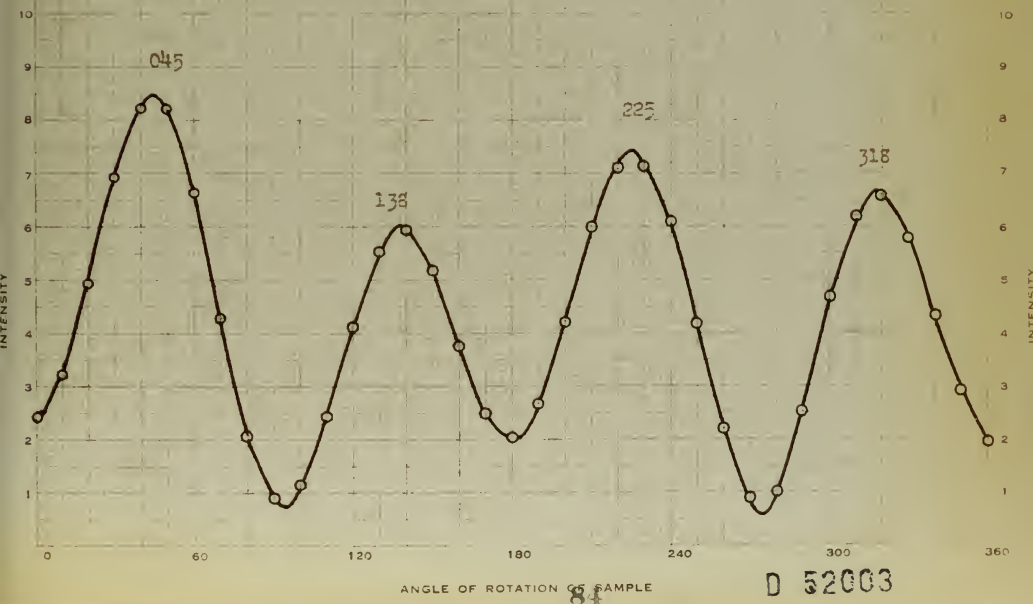
@ 045		@ 138		@ 225		@ 318	
M <sub>1</sub>	R	M	R	M <sub>3</sub>	R	M <sub>5</sub>	R <sub>4</sub>
43.1	48.1	41.1	42.7	43.7	48.0	40.8	42.3
43.1	47.3	40.8	43.0	42.3	48.2	40.8	42.7
43.10	47.95	40.95	42.85	42.50	48.10	40.80	42.75

M <sub>1</sub>	43.10	R <sub>1</sub>	47.95
M <sub>2</sub>	40.95	R <sub>2</sub>	42.85
$\Delta M_{12}$	2.15	$\Delta R_{12}$	5.10

M <sub>3</sub>	42.50	R <sub>3</sub>	48.10
M <sub>4</sub>	40.80	R <sub>4</sub>	42.75
$\Delta M_{34}$	1.70	$\Delta R_{34}$	5.35

AV  $\Delta M$  1.93 PHASE SHIFT  
 AV  $\Delta R$  5.23 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE NO 1A

6 PPM N<sub>2</sub>

DATE 5 APRIL 1952 PM

CRYSTAL NO 2

SURFACE FILM CONDITION

15th AUTOCLAVE OF 15 MIN. 233°C

@ 046		@ 140		@ 226		@ 318	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.2	48.5	43.7	43.2	44.5	48.7	42.8	42.8
44.7	48.6	43.4	43.2	44.4	48.6	42.5	43.0
AV. 45.45	48.55	43.55	43.20	44.45	48.65	42.65	42.90

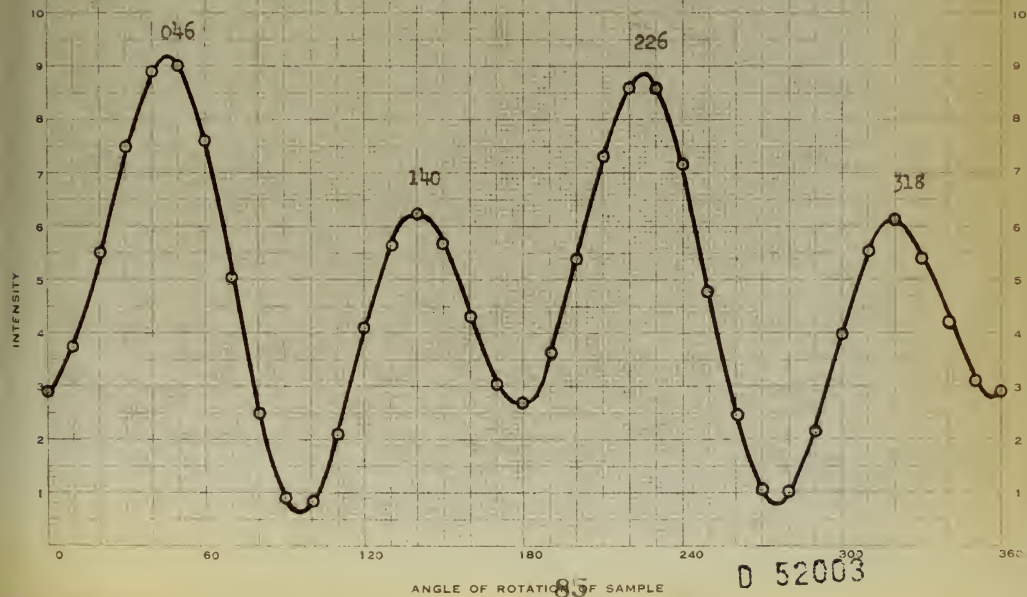
M <sub>1</sub>	45.45	R <sub>1</sub>	48.55
M <sub>2</sub>	43.55	R <sub>2</sub>	43.20
$\Delta M_{12}$	1.90	$\Delta R_{12}$	5.35

M <sub>3</sub>	44.45	R <sub>3</sub>	48.65
M <sub>4</sub>	42.65	R <sub>4</sub>	42.90
$\Delta M_{34}$	1.80	$\Delta R_{34}$	5.75

AV  $\Delta M$  1.85 PHASE SHIFT  
 AV  $\Delta R$  5.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



2M

2R

PHASE SHIFT (2m) AND  
ROTATION OF PLANE OF  
POLARIZATION (2r) VS.  
CORROSION TIME FOR  
SAMPLE NO. 1B-1

2.5  
2.0  
1.5  
1.0  
0.5  
0.0  
-0.5  
-1.0  
-1.5  
-2.0

22  
21  
20  
19  
18  
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16  
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12  
11  
10  
9  
8  
7  
6  
5  
4  
3  
2  
1  
0  
-1  
-2  
-3  
-4  
-5  
-6  
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-12  
-13  
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-15  
-16  
-17  
-18  
-19  
-20  
-21  
-22

E

1

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9

10

11

12

13

14

15

AUTOClaves AT 233°C

D 52003

D 52003





SAMPLE No. 1B

70 PPM  $N_2$ 

DATE 5 MARCH 1952 AM

CRYSTAL No. 1

SURFACE FILM CONDITION

AS ELECTROPOLISHED

@ 332°		@ 242°		@ 152°		@ 62°	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.2	46.9	39.5	44.9	45.8	46.9	39.0	44.8
46.4	46.9	39.4	45.0	45.8	46.9	38.8	44.8
AV. 46.30	46.90	39.45	44.95	45.80	46.90	38.90	44.80

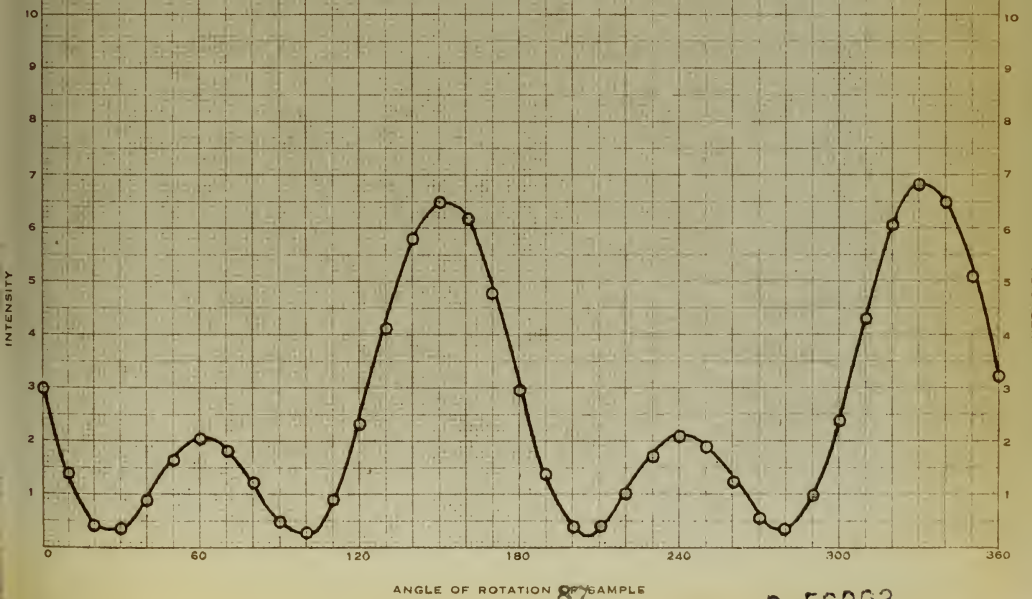
M <sub>1</sub>	46.30	R <sub>1</sub>	46.90
M <sub>2</sub>	39.45	R <sub>2</sub>	44.95
$\Delta M_{12}$	6.85	$\Delta R_{12}$	1.95

M <sub>3</sub>	45.80	R <sub>3</sub>	46.90
M <sub>4</sub>	38.90	R <sub>4</sub>	44.80
$\Delta M_{34}$	6.70	$\Delta R_{34}$	2.10

AV  $\Delta M$  6.77 PHASE SHIFTAV  $\Delta R$  2.03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





SAMPLE NO 1B

70 PPM  $N_2$ 

DATE 13 MARCH 1952

CRYSTAL NO. 1

SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. at 233°C

@ 296.....		@ 206.....		@ 116.....		@ 026....	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.2	45.9	37.7	45.2	47.9	46.0	37.7	45.0
48.0	45.9	38.0	45.2	47.9	45.8	37.5	45.0
48.10	45.90	37.85	45.20	47.90	45.90	37.60	45.00

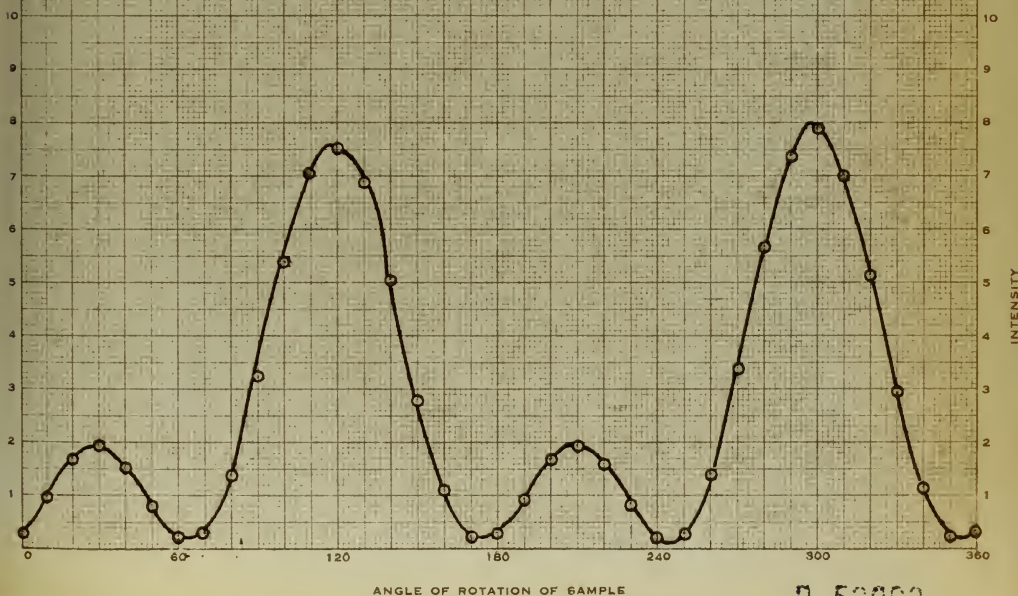
M<sub>1</sub> 48.10      R<sub>1</sub> 45.90  
 M<sub>2</sub> 37.85      R<sub>2</sub> 45.20  
 $\Delta M_{12}$  10.25       $\Delta R_{12}$  0.70

M<sub>3</sub> 47.90      R<sub>3</sub> 45.90  
 M<sub>4</sub> 37.60      R<sub>4</sub> 45.00  
 $\Delta M_{34}$  10.30       $\Delta R_{34}$  0.90

AV  $\Delta M$  10.27      PHASE SHIFTAV  $\Delta R$  0.80      PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE No. 1B

70 PPM  $N_2$ 

DATE 14 MARCH 1952 AM

CRYSTAL No. 1

SURFACE FILM CONDITION 2nd AUTOCLAVE 15 MIN. at 233°C

@ 275		@ 185		@ 95		@ 005	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
49.6	46.0	37.1	45.6	49.2	46.0	37.1	45.5
49.5	46.0	37.0	45.6	49.1	46.0	37.2	45.5
AV. 49.55	46.00	37.05	45.60	49.15	46.00	37.15	45.50

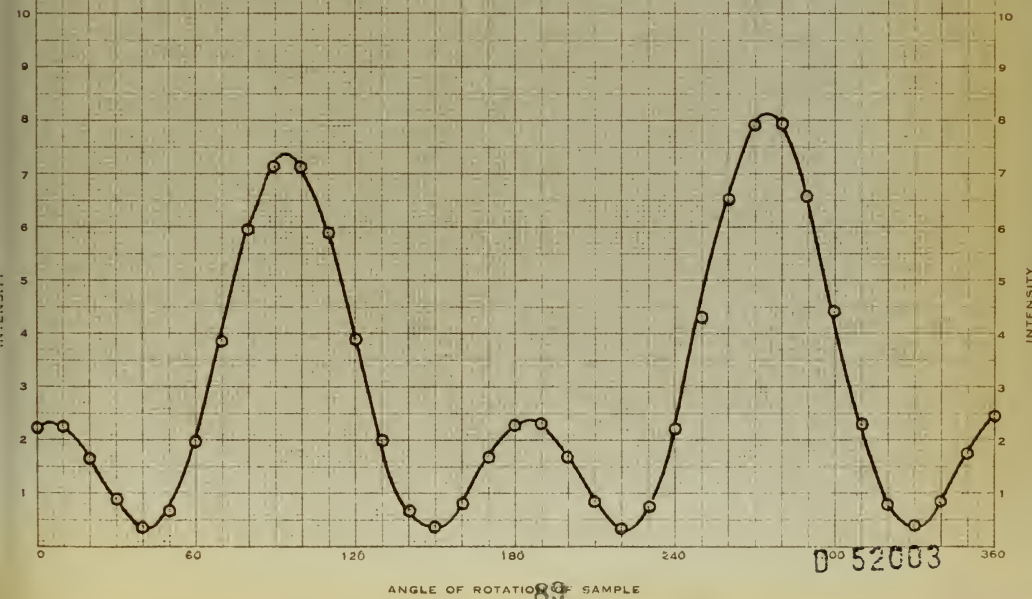
M <sub>1</sub>	49.55	R <sub>1</sub>	46.00
M <sub>2</sub>	37.05	R <sub>2</sub>	45.60
$\Delta M_{12}$	12.50	$\Delta R_{12}$	0.40

M <sub>3</sub>	49.15	R <sub>3</sub>	46.00
M <sub>4</sub>	37.15	R <sub>4</sub>	45.50
$\Delta M_{34}$	12.00	$\Delta R_{34}$	0.50

AV  $\Delta M$  12.25 PHASE SHIFTAV  $\Delta R$  0.45 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



0 52003

ANGLE OF ROTATION OF SAMPLE





CRYSTAL No 1 SURFACE FILM CONDITION 3rd AUTOCLAVE 15 MIN. at 233°C

② 202		④ 202		⑥ 112		⑧ 022	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.1	45.6	36.0	46.0	49.8	46.0	35.9	45.8
49.5	45.8	35.9	46.0	49.5	46.0	35.8	45.3
48.9	45.8						
48.7	45.8						
48.8	45.8						
49.20	45.20	35.95	46.0	49.65	46.0	35.85	45.20

AV

M <sub>1</sub>	49.20	R <sub>1</sub>	45.80
M <sub>2</sub>	35.95	R <sub>2</sub>	46.00
$\Delta M_{12}$	13.25	$\Delta R_{12}$	-0.20

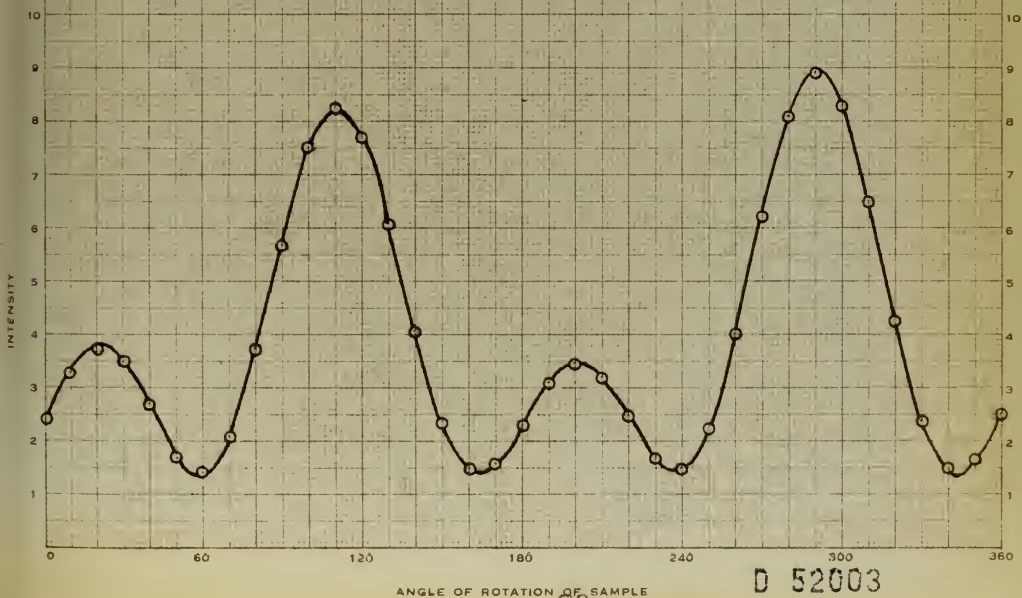
M <sub>3</sub>	49.65	R <sub>3</sub>	46.00
M <sub>4</sub>	35.85	R <sub>4</sub>	45.80
$\Delta M_{34}$	13.40	$\Delta R_{34}$	0.20

AV  $\Delta M$  13.32 PHASE SHIFT

AV  $\Delta R$  0.0 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE No 11

70 PPM

DATE 20 MAR 31 1957 AM

CRYSTAL No.

SURFACE FILM CONDITION

15 MIN. @ 233°C

@ 012		@ 102		@ 192		@ 282	
M <sub>1</sub>	R	M <sub>2</sub>	R <sub>2</sub>	M	R <sub>1</sub>	M <sub>3</sub>	R <sub>4</sub>
47.8	45.3	35.0	45.5	48.8	45.2	35.0	45.5
48.6	45.5	34.9	45.6	48.6	45.4	34.7	45.7
Av. 48.70	45.40	34.9	45.6	48.70	45.30	34.85	45.60

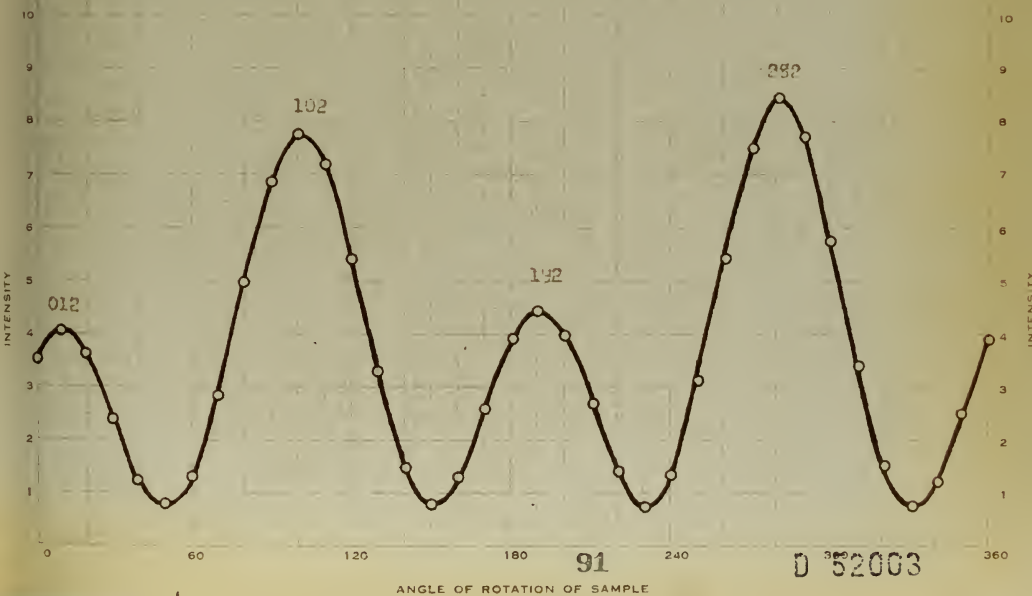
M <sub>1</sub> 48.70	R 45.40
M <sub>2</sub> 34.90	R <sub>2</sub> 45.60
$\Delta M_{12}$ 13.80	$\Delta R_{12}$ -0.20

M <sub>3</sub> 48.70	R <sub>3</sub> 45.30
M <sub>4</sub> 34.85	R <sub>4</sub> 45.60
$\Delta M_{34}$ 13.85	$\Delta R_{34}$ -0.30

AV  $\Delta M$  13.82 PHASE SHIFT  
 AV  $\Delta R$  -0.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE NO 13

10 PPM  $H_2$ 

DATE 24 MARCH 1952 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

5th AUTOCLAVE 15 MIN. @ 233°C

@ 254		@ 174		@ 074		@ 344	
M1	R1	M2	R2	M3	R3	M4	R4
49.9	45.2	33.6	45.8	49.3	45.1	33.5	45.8
43.6	45.2	33.5	45.9	49.6	45.2	33.6	45.8
AV 49.75	45.20	33.55	45.85	49.45	45.10	33.55	45.80

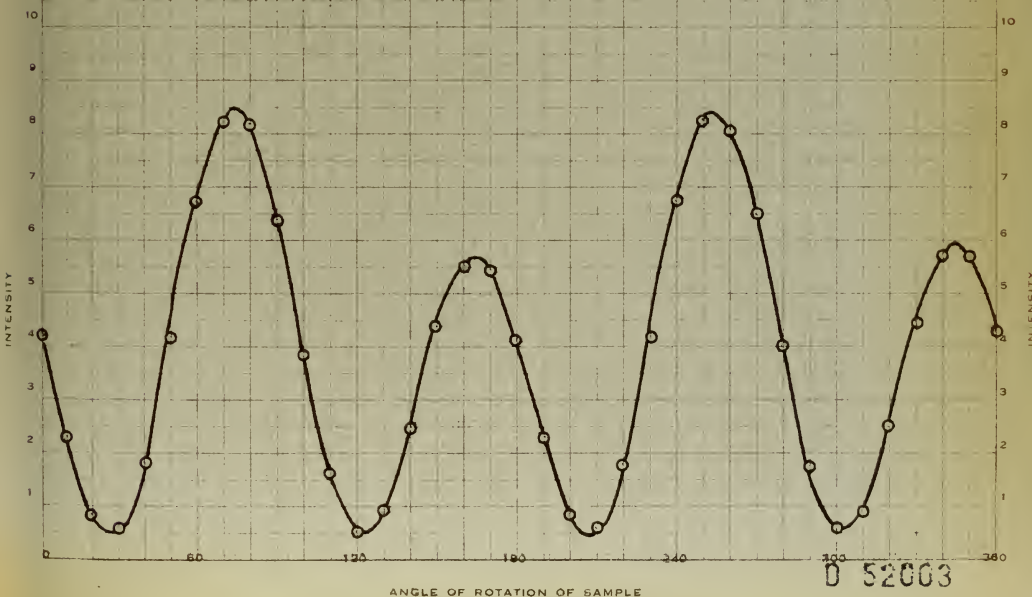
$M_1$  49.75       $R_1$  45.20  
 $M_2$  33.55       $R_2$  45.85  
 $\Delta M_{12}$  16.20       $\Delta R_{12}$  -0.65

$M_3$  49.45       $R_3$  45.10  
 $M_4$  33.55       $R_4$  45.80  
 $\Delta M_{34}$  15.90       $\Delta R_{34}$  -0.70

AV  $\Delta M$  16.05 PHASE SHIFTAV  $\Delta R$  -0.68 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 13

70 PPM  $N_2$ 

DATE 25 MARCH 1952

CRYSTAL NO 1

SURFACE FILM CONDITION

6th AUTOCLAVE 15 MIN. @ 233°C

@ 356		@ 356		@ 176		@ 086	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.3	45.2	35.0	46.0	51.5	45.0	35.4	46.0
51.5	45.0	34.8	46.0	51.3	44.8	34.9	46.1
						34.7	46.0
AV. 51.40	45.10	34.90	46.00	51.40	44.90	35.00	46.03

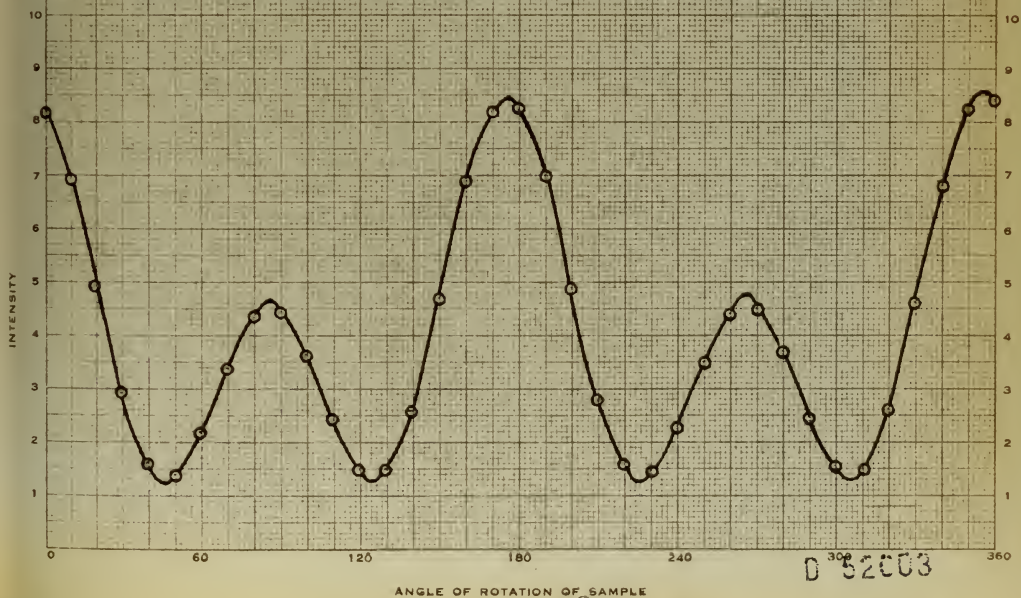
M <sub>1</sub>	51.40	R <sub>1</sub>	45.10
M <sub>2</sub>	34.90	R <sub>2</sub>	46.00
$\Delta M_{12}$	16.50	$\Delta R_{12}$	-0.90

M <sub>3</sub>	51.40	R <sub>3</sub>	44.90
M <sub>4</sub>	35.00	R <sub>4</sub>	46.03
$\Delta M_{34}$	16.40	$\Delta R_{34}$	-1.13

AV  $\Delta M$  16.45 PHASE SHIFTAV  $\Delta R$  -1.01 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE No 1B

70 PPM  $H_2$ 

DATE 26 MARCH 1952 PM

CRYSTAL No 1

SURFACE FILM CONDITION

7th AUTOCLAVE 15 MIN. @ 233°C

@ 305		@ 216		@ 126		@ 056	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.8	44.9	34.2	46.1	52.2	44.8	33.9	46.1
52.7	44.7	34.0	46.2	52.1	44.8	33.7	46.1
52.75	44.80	34.10	46.15	52.15	44.80	33.80	46.10

M<sub>1</sub> 52.75      R<sub>1</sub> 44.80  
 M<sub>2</sub> 34.10      R<sub>2</sub> 46.15  
 ΔM<sub>12</sub> 18.65      ΔR<sub>12</sub> -1.35

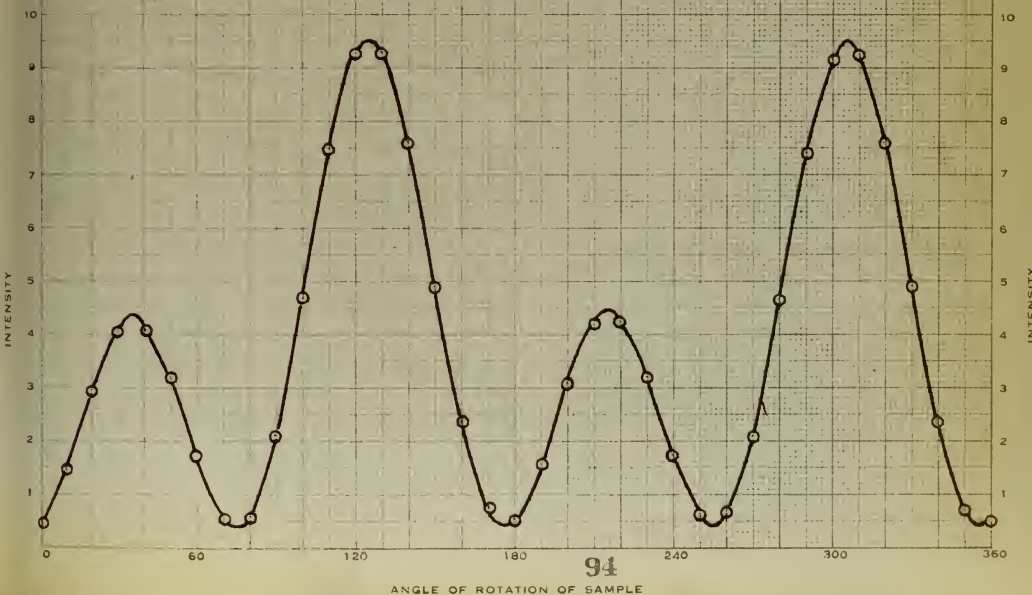
M<sub>3</sub> 52.15      R<sub>3</sub> 44.80  
 M<sub>4</sub> 33.80      R<sub>4</sub> 46.10  
 ΔM<sub>34</sub> 18.35      ΔR<sub>34</sub> -1.30

Av ΔM 18.50 PHASE SHIFT

Av ΔR -1.33 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE No. 13

70 PPM  $N_2$ 

DATE 27 MARCH 1952 AM

CRYSTAL No. 1

SURFACE FILM CONDITION

8th AUTOCLAVE 15 MIN. @ 233°C

@ 325

@ 235

@ 148

@ 052

M<sub>1</sub>R<sub>1</sub>M<sub>2</sub>R<sub>2</sub>M<sub>3</sub>R<sub>3</sub>M<sub>4</sub>R<sub>4</sub>

53.2

44.7

33.6

46.1

52.7

44.8

33.2

46.2

53.1

44.8

33.4

46.3

52.7

44.7

33.5

46.3

AV.

53.15

44.75

33.50

46.20

52.70

44.75

33.35

46.25

M<sub>1</sub> 53.15R<sub>1</sub> 44.75M<sub>2</sub> 33.50R<sub>2</sub> 46.20 $\Delta M_{12}$  19.65 $\Delta R_{12}$  -1.45M<sub>3</sub> 52.70R<sub>3</sub> 44.75M<sub>4</sub> 33.35R<sub>4</sub> 46.25 $\Delta M_{34}$  19.35 $\Delta R_{34}$  -1.50AV  $\Delta M$  19.50

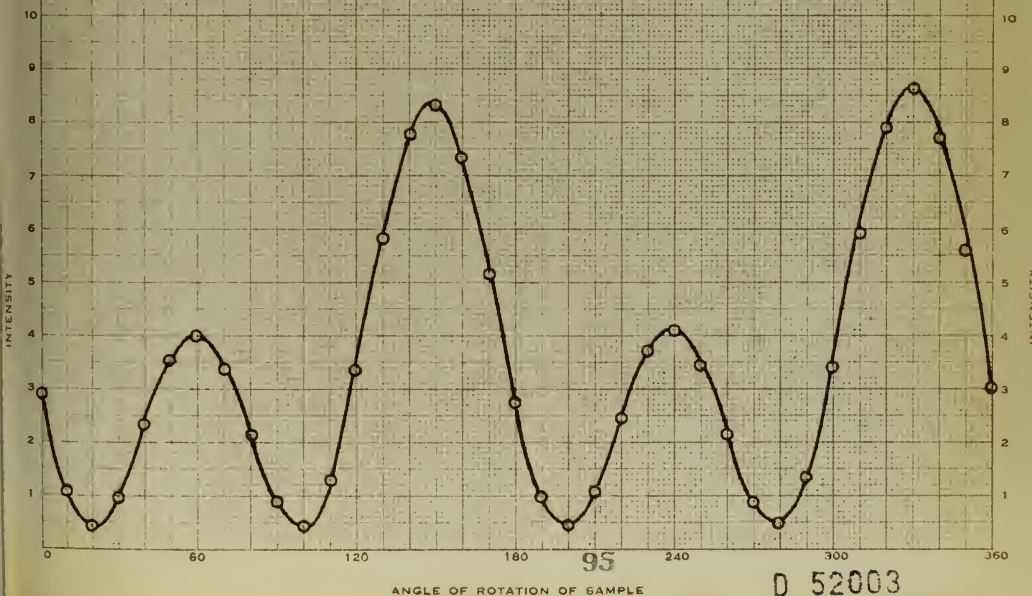
PHASE SHIFT

AV  $\Delta R$  -1.47

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE No 1B

70 PPM N<sub>2</sub>

DATE 28 MARCH 1952 AM

CRYSTAL No. 1

SURFACE FILM CONDITION 9th AUTOCLAVE 15 MIN. @ 233°C

@ 205.....		@ 215.....		@ 225.....		T	@ 295.....	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>		M <sub>4</sub>	R <sub>4</sub>
56.2	44.4	34.2	46.6	55.5	45.0		34.3	46.5
56.5	44.6	34.2	46.5	55.8	44.8		34.1	46.6
Av. 56.35	44.50	34.20	46.55	55.65	44.90		34.20	46.55

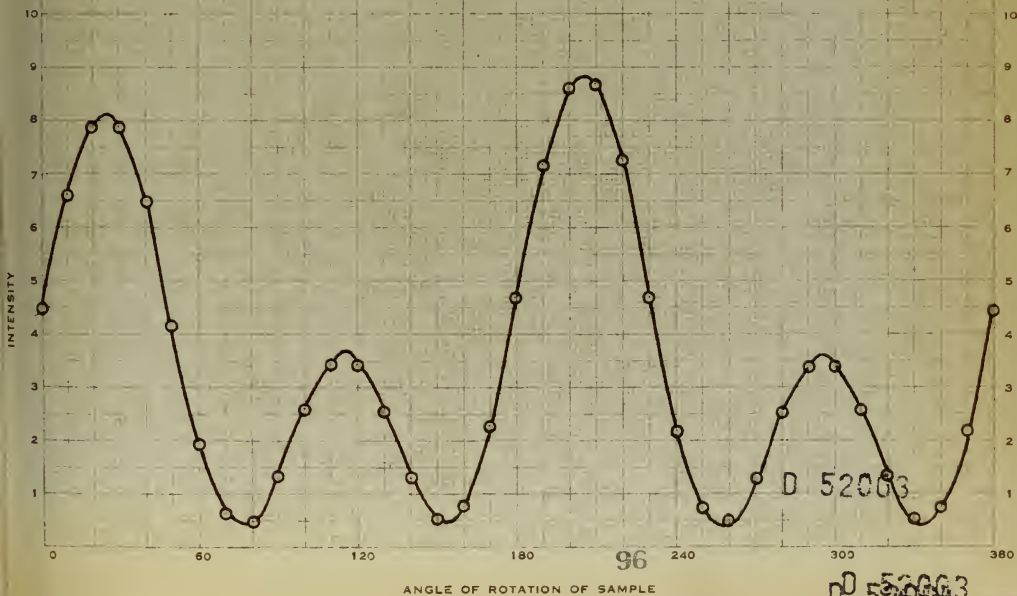
M <sub>1</sub>	56.35	R <sub>1</sub>	44.50
M <sub>2</sub>	34.20	R <sub>2</sub>	46.55
$\Delta M_{12}$	22.15	$\Delta R_{12}$	-2.05

M <sub>3</sub>	55.65	R <sub>3</sub>	44.90
M <sub>4</sub>	34.20	R <sub>4</sub>	46.55
$\Delta M_{34}$	21.45	$\Delta R_{34}$	-1.65

Av  $\Delta M$  21.80 PHASE SHIFTAv  $\Delta R$  -1.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







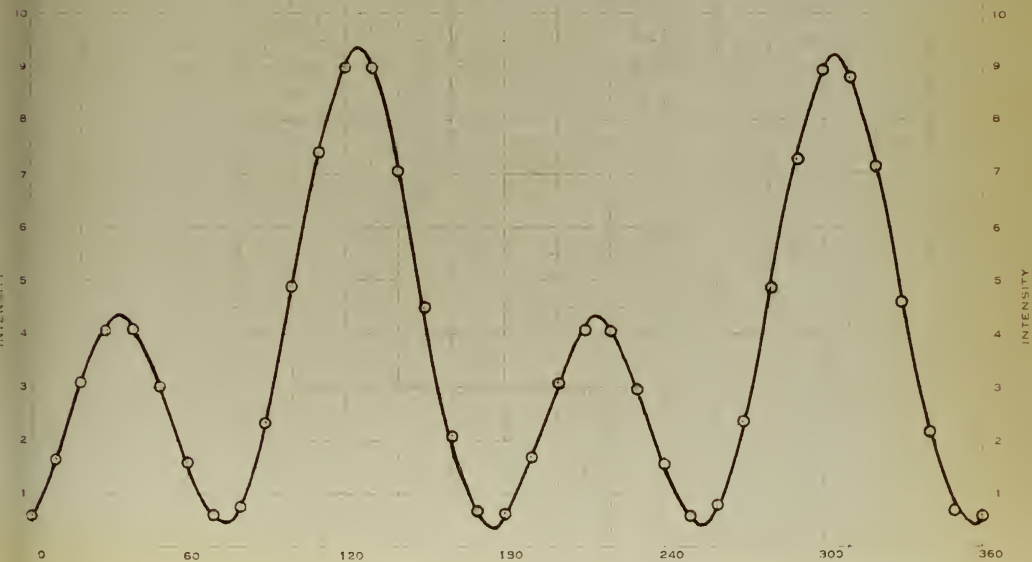
@ 304		@ 214		@ 124		@ 034	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
55.6	44.9	34.0	46.2	55.7	45.0	33.6	46.4
55.5	45.1	34.0	46.3	55.8	45.1	33.9	46.3
55.55	45.00	34.00	46.25	55.75	45.05	33.75	46.35

M	55.55	R	45.00
M <sub>2</sub>	34.00	R <sub>2</sub>	46.25
ΔM <sub>2</sub>	21.55	ΔR <sub>2</sub>	-1.25

M <sub>3</sub>	55.75	R <sub>3</sub>	45.05
M <sub>4</sub>	33.75	R <sub>4</sub>	46.35
ΔM <sub>3,4</sub>	22.00	ΔR <sub>3,4</sub>	-1.30

AV ΔM 21.77 PHASE SHIFT  
 AV ΔR -1.26 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 1B

30 PPM  $\lambda_D$ 

DATE 1 APRIL 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION

11th AUTOCLAVE 15 MIN. @ 233°C

@ 353		@ 243		@ 153		@ 063	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.4	45.0	33.2	46.6	52.4	45.1	32.4	46.2
52.3	44.9	32.9	46.5	52.6	45.2	32.8	46.3
52.35	44.95	33.05	46.55	52.50	45.15	32.60	46.25

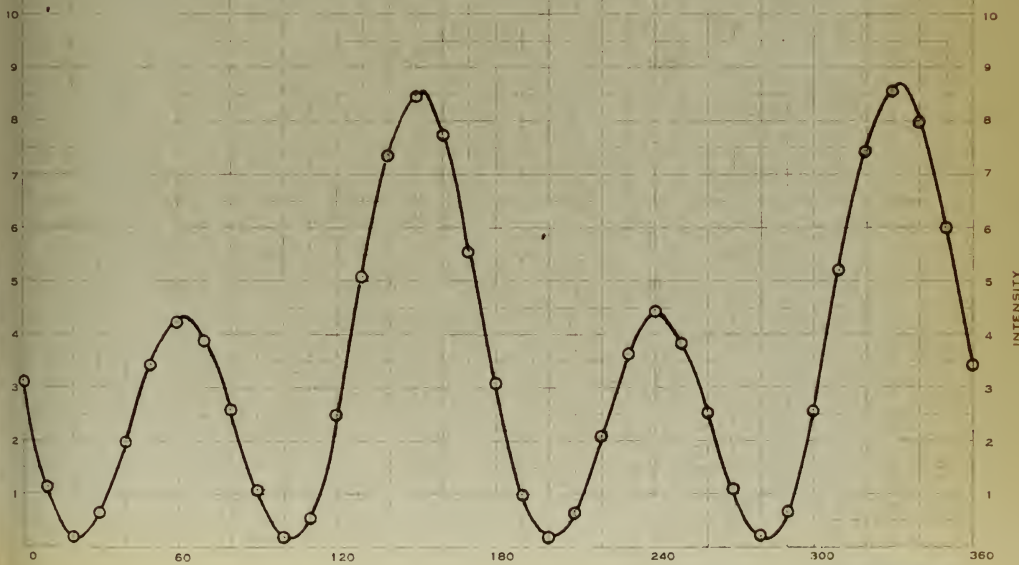
M <sub>1</sub>	52.35	R <sub>1</sub>	44.95
M <sub>2</sub>	33.05	R <sub>2</sub>	46.55
$\Delta M_{12}$	19.30	$\Delta R_{12}$	-1.60

M <sub>3</sub>	52.50	R <sub>3</sub>	45.15
M <sub>4</sub>	32.60	R <sub>4</sub>	46.25
$\Delta M_{34}$	19.90	$\Delta R_{34}$	-1.10

AV  $\Delta M$  19.60 PHASE SHIFTAV  $\Delta R$  -1.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



98 D 52003

DD 520043



SAMPLE NO 1B

90 PPM  $N_2$ 

DATE 4 APRIL 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION

13th AUTOCLAVE 15 MIN. @ 233°C

@ 214		@ 124		@ 034		@ 304	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.6	45.4	30.9	46.1	52.3	45.3		
52.6	45.6	31.2	46.4				
52.60	45.50	31.15	46.25				

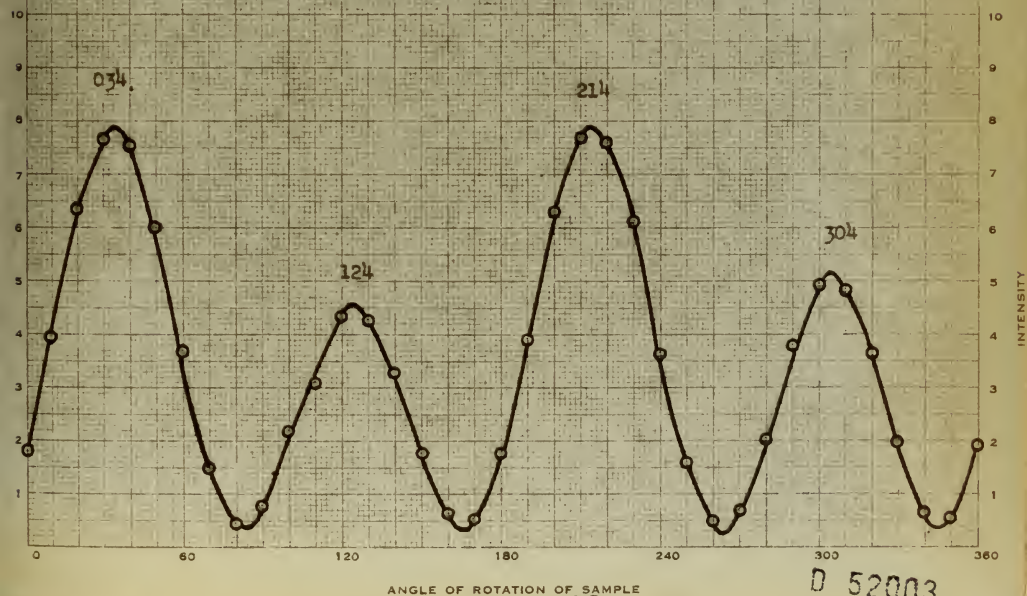
M <sub>1</sub>	52.60	R <sub>1</sub>	45.50
M <sub>2</sub>	31.15	R <sub>2</sub>	46.25
$\Delta M_{12}$	21.45	$\Delta R_{12}$	-.75

M <sub>3</sub>		R <sub>3</sub>	
M <sub>4</sub>		R <sub>4</sub>	
$\Delta M_{34}$		$\Delta R_{34}$	

 $\Delta V$   $\Delta M$  PHASE SHIFT $\Delta V$   $\Delta R$  PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







CRYSTAL No 1

SURFACE FILM CONDITION

124h ATTACHMENT 15 MIN. @ 233°C

AV

@ 253		@ 163		@ 073		@ 343	
M	R	M	R	M	R	M	R
53.6	44.9	32.5	46.6	53.4	44.8	32.9	46.3
53.5	45.1	32.8	46.4	53.2	45.0	32.7	46.6
53.55	45.00	32.65	46.50	53.30	44.90	32.80	46.55

M	53.55	R	45.00
M	32.65	R	46.50
$\Delta M$	20.50	$\Delta R$	-1.65

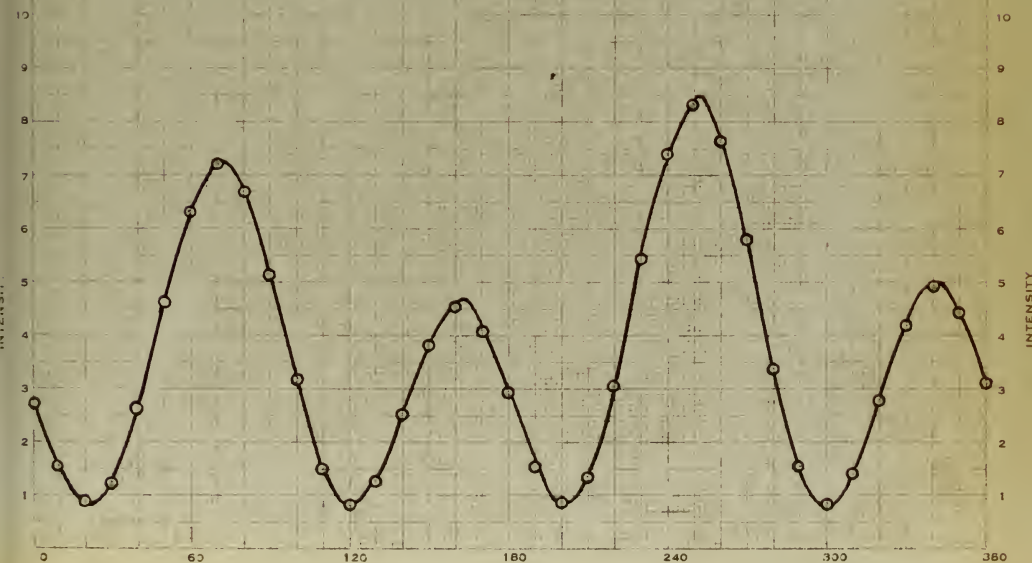
M	53.30	R	44.90
M	32.80	R	46.55
$\Delta M_{34}$	20.50	$\Delta R_{34}$	-1.65

AV  $\Delta M$  20.70 PHASE SHIFT

AV  $\Delta R$  -1.57 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



SAMPLE NO. 1B

70 PPM  $N_2$ 

DATE 5 APRIL 1952 AM

CRYSTAL No. 1

SURFACE FILM CONDITION

15th AUTOCLAVE OF 15 MIN. @ 233°C

@ ... 354 ...		@ ... 244 ...		@ ... 154 ...		@ ... 064 ...	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
55.7	45.1	34.3	45.7	54.5	45.3	34.2	46.0
55.6	44.9	34.2	45.6	54.7	45.2	34.0	45.8
55.65	45.00	34.25	45.60	54.50	45.25	34.10	45.90

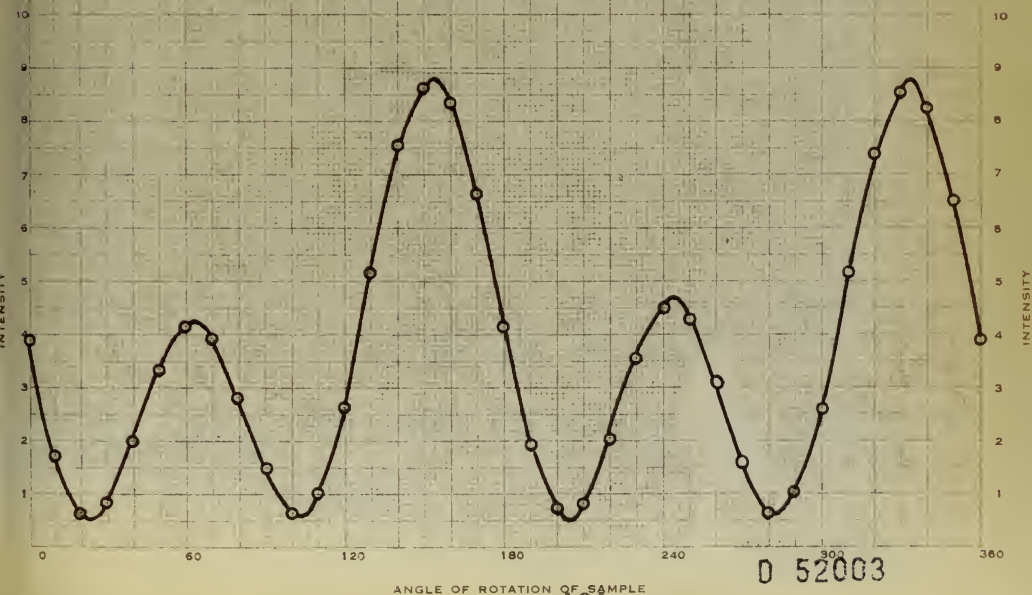
M<sub>1</sub> 55.65      R<sub>1</sub> 45.00  
 M<sub>2</sub> 34.25      R<sub>2</sub> 45.80  
 $\Delta M_{12}$  21.40       $\Delta R_{12}$  - .80

M<sub>3</sub> 54.50      R<sub>3</sub> 45.25  
 M<sub>4</sub> 34.10      R<sub>4</sub> 45.90  
 $\Delta M_{34}$  20.40       $\Delta R_{34}$  - .65

AV  $\Delta M$  20.90 PHASE SHIFTAV  $\Delta R$  -0.72 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



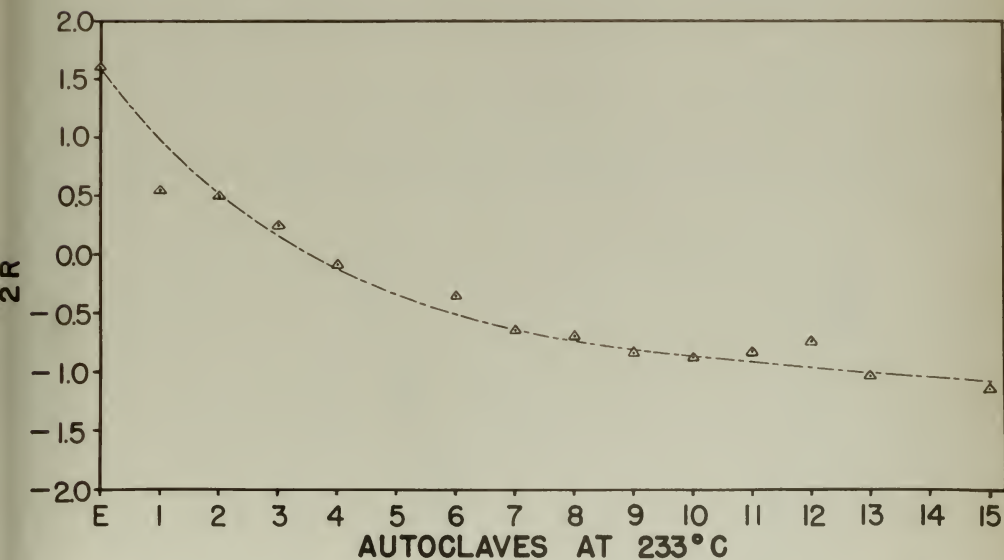
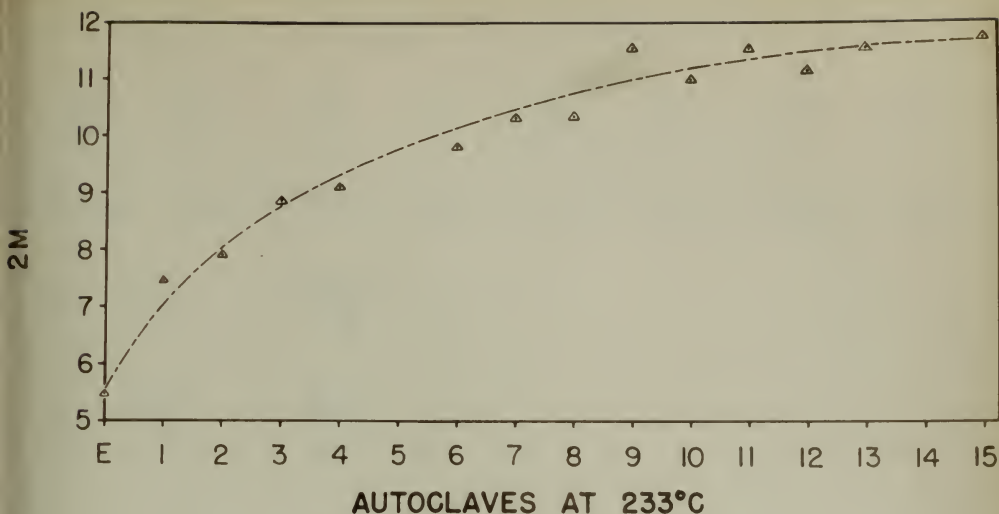
D 52003

ANGLE OF ROTATION OF SAMPLE

161







PHASE SHIFT (2M) AND ROTATION OF PLANE OF  
POLARIZATION (2R) VS. CORROSION TIME FOR  
SAMPLE NO. 1C-1



SAMPLE NO 1C

113 FPM  $H_2$

DATE 5 March 1962 PM

CRYSTAL NO 1

SURFACE FILM CONDITION AS ELECTROPOLISHED

@ 306		@ 036		@ 126		@ 216	
M	R	M <sub>2</sub>	R	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.5	46.6	39.2	45.0	45.5	46.7	39.8	44.9
44.8	46.7	39.6	45.2	45.3	46.7	39.6	45.2
44.65	46.65	39.40	45.10	45.40	46.70	39.70	45.05

M	44.65	R	46.65
M <sub>2</sub>	39.40	R <sub>2</sub>	45.10
$\Delta M_{12}$	5.25	$\Delta R_{12}$	1.55

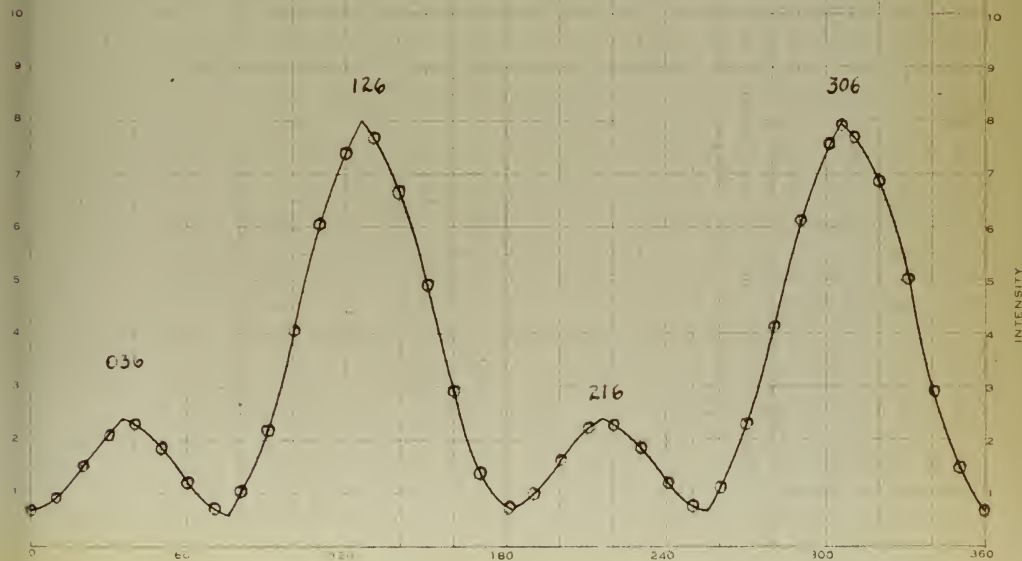
M <sub>3</sub>	45.40	R <sub>3</sub>	46.70
M <sub>4</sub>	39.70	R <sub>4</sub>	45.05
$\Delta M_{34}$	5.70	$\Delta R_{34}$	1.65

AV  $\Delta M$  5.43 PHASE SHIFT

AV  $\Delta R$  1.60 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 10

113 PPM  $K_2$ 

DATE 12 March 1952 PM

CRYSTAL NO 1

SURFACE FILM COMPOSITION

1st AUTOCLAVE 15 Min at 233°C

@ 306		@ 036		@ 126		@ 216	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.5	45.9	39.0	45.1	45.8	45.8	39.7	45.0
45.4	45.7	38.0	45.4	45.7	45.5	37.9	45.2
45.45	45.80	38.00	45.25	45.75	45.65	38.30	45.10

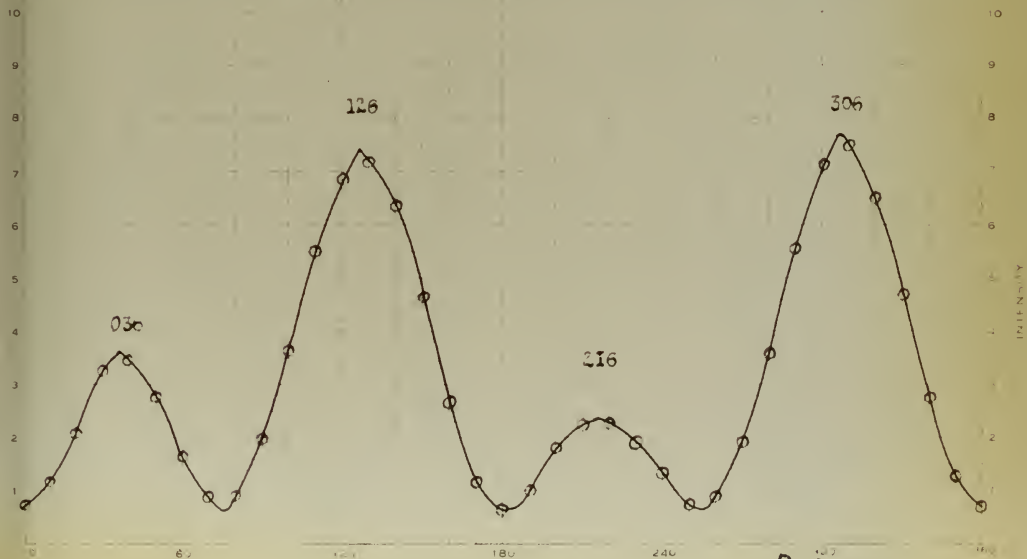
M <sub>1</sub>	45.45	R <sub>1</sub>	45.80
M <sub>2</sub>	39.00	R <sub>2</sub>	45.25
$\Delta M_{12}$	7.45	$\Delta R_{12}$	0.55

M <sub>3</sub>	45.75	R <sub>3</sub>	45.65
M <sub>4</sub>	38.30	R <sub>4</sub>	45.10
$\Delta M_{34}$	7.45	$\Delta R_{34}$	0.55

AV  $\Delta M$  7.45 PHASE SHIFTAV  $\Delta R$  0.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

104

D 52003





SAMPLE No. 10 113 PPM 105.2 PM

CRYSTAL No. 1 SURFACE FILM CONDITION 10 min. at 233°C

@ 0°		@ 15°		@ 30°		@ 33°	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.0	46.2	37.9	45.7	40.2	40.1	37.3	45.6
45.0	43.0	38.0	45.5	40.3	40.3	38.7	45.2
46.00	46.10	37.95	45.60	40.25	40.20	37.00	45.90

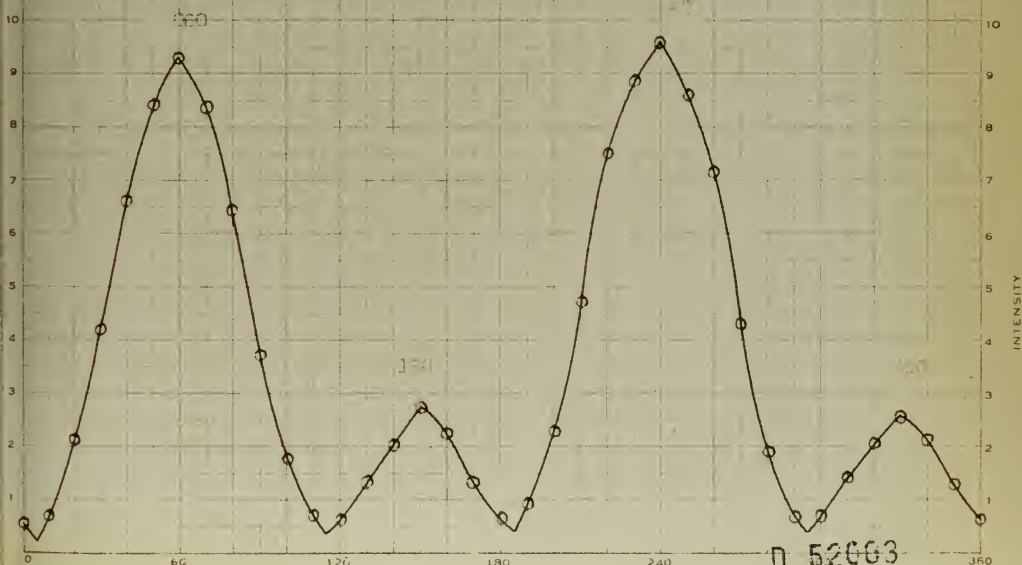
M <sub>1</sub>	46.00	R <sub>1</sub>	46.10
M <sub>2</sub>	37.95	R <sub>2</sub>	45.60
ΔM <sub>12</sub>	8.05	ΔR <sub>12</sub>	.50

M <sub>3</sub>	40.25	R <sub>3</sub>	40.20
M <sub>4</sub>	38.75	R <sub>4</sub>	45.70
ΔM <sub>34</sub>	7.75	ΔR <sub>34</sub>	.50

Av ΔM 7.30 PHASE SHIFT  
Av ΔR .50 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE



CRYSTAL No

SURFACE FILM CONDITION

@ 15		@ 30		@ 50		@ 75	
M <sub>1</sub>	R	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>1</sub>	M <sub>4</sub>	R <sub>4</sub>
41.4	43.1	40.7	44.4	47.1	43.9	39.7	47.8
47.0	39.0	41.2	45.7	47.3	43.7	39.1	48.5
ΔM <sub>12</sub>	6.70	ΔM <sub>23</sub>	6.55	ΔM <sub>34</sub>	6.70	ΔM <sub>41</sub>	6.70

M <sub>1</sub>	43.00	R <sub>1</sub>	41.40
M <sub>2</sub>	40.10	R <sub>2</sub>	45.35
ΔM <sub>12</sub>	2.90	ΔR <sub>12</sub>	3.95

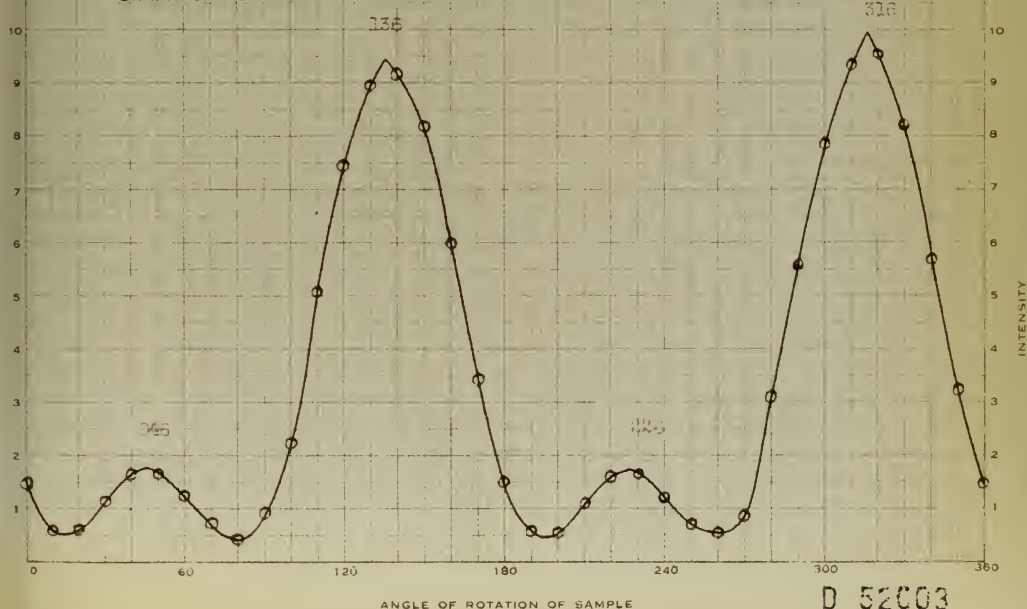
M <sub>3</sub>	47.05	R <sub>3</sub>	43.10
M <sub>4</sub>	46.05	R <sub>4</sub>	43.75
ΔM <sub>34</sub>	0.90	ΔR <sub>34</sub>	0.60

AV ΔM 2.5 PHASE SHIFT

AV ΔR 2.5 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



SAMPLE NO. 12

113 PPM 12

DATE 22 MARCH 1968 JN

CRYSTAL NO. 1

SURFACE FILM CONDITION

164 517 1100

33 127 1 5 2100

@ 229		@ 113		@ 109		@ 102	
M1	R1	M	R	M3	R1	M1	R1
46.4	45.3	37.1	45.1	46.0	45.2	37.2	45.2
46.2	45.1	37.2	45.5	46.0	45.1	37.3	45.2
Av 46.30	45.00	37.15	45.30	46.00	45.15	37.00	45.20

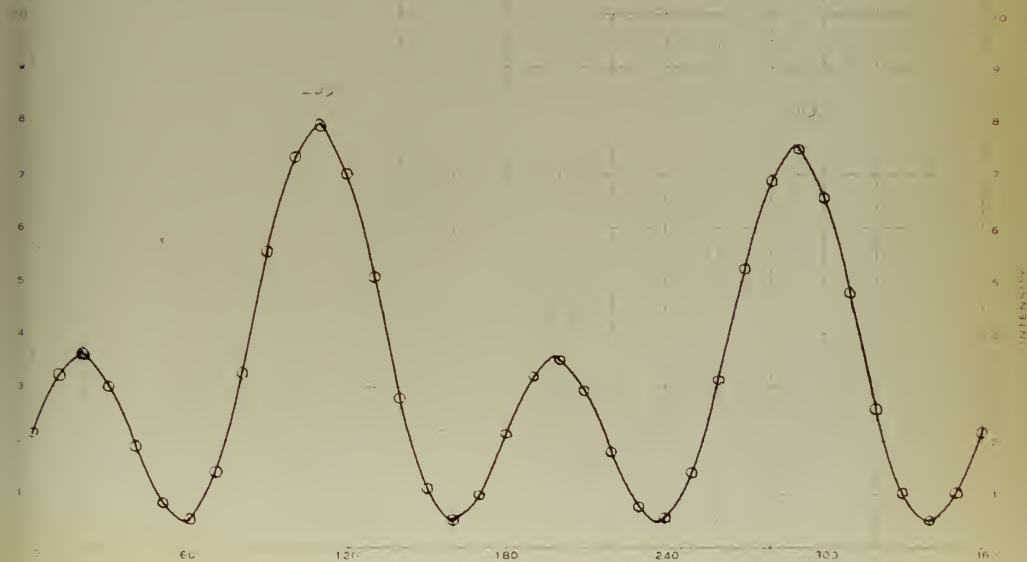
M	46.30	R	45.30
M	37.15	R	45.30
$\Delta M$	9.15	$\Delta R$	- .10

M	46.00	R	45.15
M	37.00	R	45.20
$\Delta M$	9.00	$\Delta R$	- .05

Av  $\Delta M$  9.08 PHASE SHIFTAv  $\Delta R$  -.08 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



CRYSTAL NO.

SURFACE FILM CONDITION

AVG CR 1-10% at 233°C

@ 141		@ 173		@ 183		@ 253	
M	R	M <sub>1</sub>	R <sub>1</sub>	M <sub>11</sub>	R <sub>11</sub>	M <sub>1</sub>	R <sub>1</sub>
42.5	45.5	38.6	45.7	47.9	45.4	38.2	45.7
45.1	45.5	38.4	46.0	47.3	45.5	38.2	46.0
Av 44.5	45.5	38.50	45.85	47.55	45.45	38.20	45.85

M	44.15	R	45.55
M	38.30	R	45.85
ΔM	4.35	ΔR	-.30

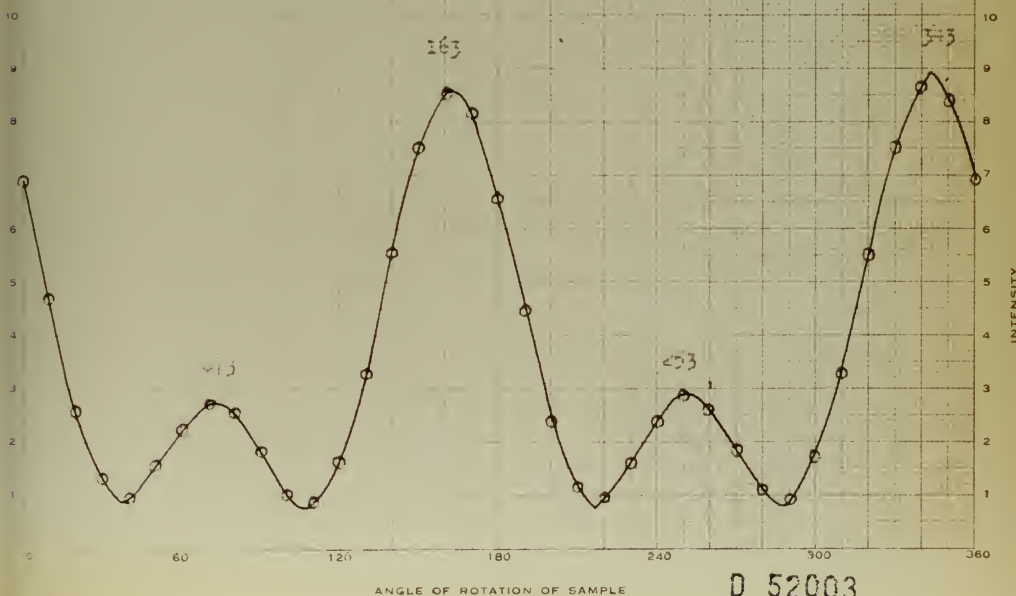
M	47.35	R	45.15
M <sub>1</sub>	38.25	R <sub>1</sub>	45.85
ΔM <sub>1</sub>	9.85	ΔR <sub>1</sub>	-.40

Av ΔM 9.80 PHASE SHIFT

Av ΔR -.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL V SURFACE FILM CONDITION

@ 335		@ 065		@ 135		@ 245	
M	R	M	R	M	R	M	R
48.5	45.2	38.0	45.3	48.1	45.2	37.6	45.7
48.4	45.1	38.2	45.7	48.1	45.1	38.1	46.0
48.45	45.15	38.10	45.75	48.10	45.15	37.85	45.85

M	48.45	R	45.15
M <sub>2</sub>	38.10	R	45.75
ΔM	10.35	ΔR	-.60

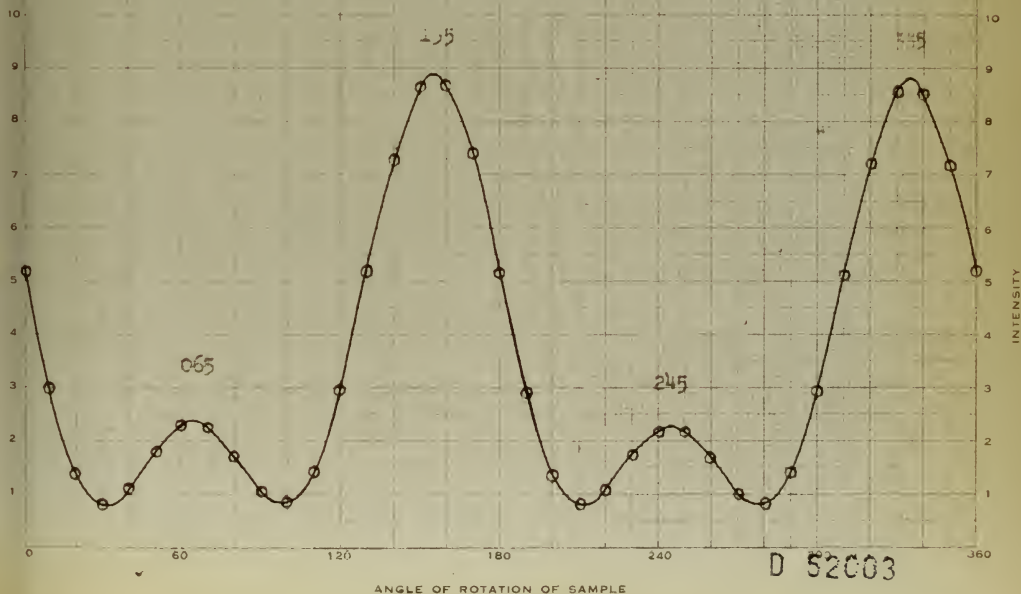
M <sub>3</sub>	48.10	R <sub>3</sub>	45.15
M <sub>4</sub>	37.85	R <sub>4</sub>	45.85
ΔM <sub>34</sub>	10.25	ΔR <sub>34</sub>	-.70

AV ΔM 10.30 PHASE SHIFT

AV ΔR -.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52C03



SAMPLE NO 10

10:00PM 8/1

DATE 7/14/61 10:00 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

8th OCTAVE OF 1.57.0 33.0

@ 287		@ 017		@ 107		@ 197	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.0	45.0	37.3	45.7	47.7	44.8	37.7	45.2
48.0	45.0	37.3	45.8	47.5	44.9	37.6	45.8
48.00	45.00	37.30	45.75	47.60	44.85	37.65	45.50

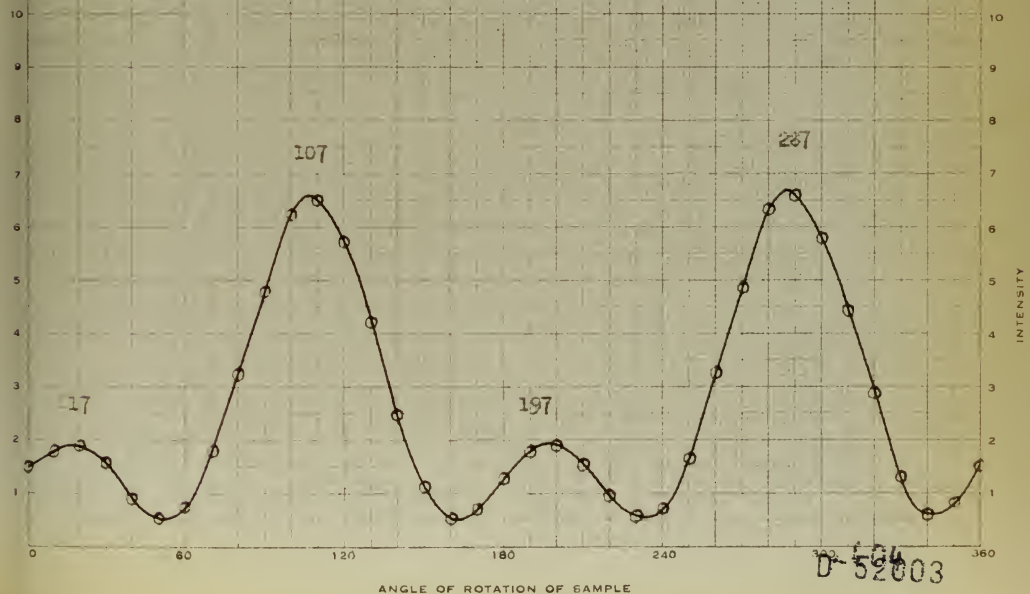
M <sub>1</sub>	48.00	R <sub>1</sub>	45.00
M <sub>2</sub>	37.30	R <sub>2</sub>	45.75
$\Delta M_{12}$	10.70	$\Delta R_{12}$	-.75

M <sub>3</sub>	47.60	R <sub>3</sub>	44.85
M <sub>4</sub>	37.65	R <sub>4</sub>	45.50
$\Delta M_{34}$	9.95	$\Delta R_{34}$	-.65

AV  $\Delta M$  10.33 PHASE SHIFTAV  $\Delta R$  -.70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D-52003



CRYSTAL NO. 1 SURFACE FILM CONDITION 5th AUTOCLAVE OF 15 MIN. @ 213°C

@ 086		@ 176		@ 266		@ 356	
M <sub>1</sub>	R	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R	M <sub>4</sub>	R <sub>4</sub>
49.6	45.0	38.2	45.8	49.0	44.9	37.4	46.0
49.0	45.2	37.6	46.0	49.2	45.1	37.4	45.8
49.30	45.10	37.90	45.90	49.10	45.00	37.40	45.90

M <sub>1</sub> 49.30	R <sub>1</sub> 45.10
M <sub>2</sub> 37.90	R <sub>2</sub> 45.90
$\Delta M_{12}$ 11.40	$\Delta R_{12}$ - .80

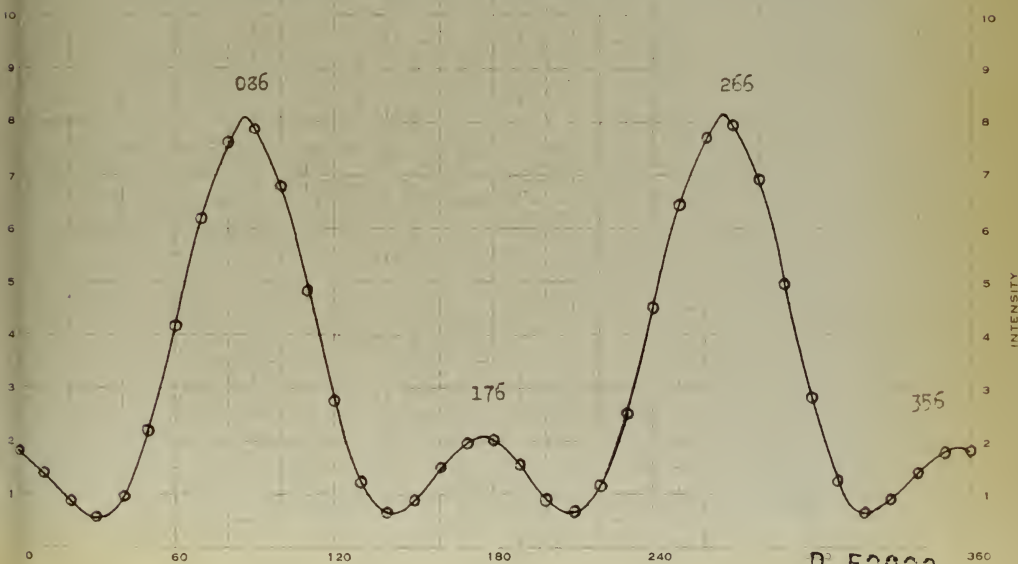
M <sub>3</sub> 49.10	R <sub>3</sub> 45.00
M <sub>4</sub> 37.40	R <sub>4</sub> 45.90
$\Delta M_{34}$ 11.70	$\Delta R_{34}$ - .90

AV  $\Delta M$  11.55 PHASE SHIFT

AV  $\Delta R$  - .85 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL NO 1

SURFACE FILM CONDITION

10th AUTOCLAVE OF 15 MIN. @ 233°C

@ 056		@ 146		@ 236		@ 326	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
49.1	45.2	37.5	46.4	48.8	44.9	38.0	45.8
49.0	45.4	37.9	46.2	48.4	45.3	38.0	45.9
49.05	45.30	37.70	46.30	48.60	45.10	38.00	45.85

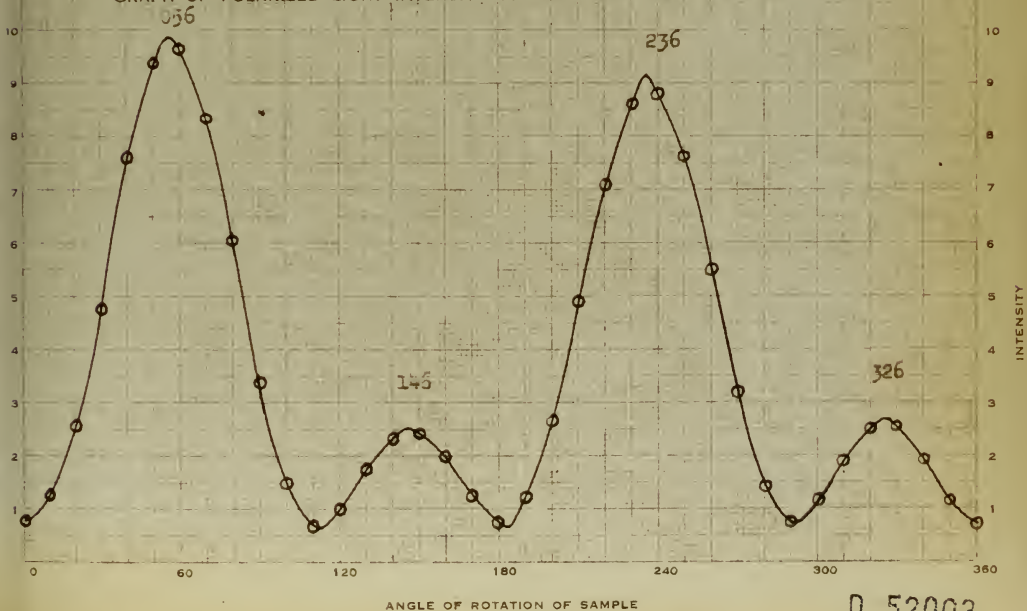
M <sub>1</sub> 49.05	R <sub>1</sub> 45.30
M <sub>2</sub> 37.70	R <sub>2</sub> 46.30
$\Delta M_{12}$ 11.35	$\Delta R_{12}$ -1.00

M <sub>3</sub> 48.60	R <sub>3</sub> 45.10
M <sub>4</sub> 38.00	R <sub>4</sub> 45.85
$\Delta M_{34}$ 10.60	$\Delta R_{34}$ - .75

AV  $\Delta M$  10.98 PHASE SHIFTAV  $\Delta R$  -.88 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 10

113 PH: N<sub>2</sub>

DATE 1 APRIL 1958 FR

CRYSTAL NO 1

SURFACE FILM CONDITION

11th AUTOCLAVE OF 15 MIN. @ 233°C

@ 335		@ 245		@ 157		@ 067	
M	R	M	R	M	R	M	R
47.0	45.0	36.1	45.7	47.3	45.0	36.1	46.1
47.8	45.0	36.1	46.0	47.6	45.2	36.3	45.9
AV 47.90	45.00	36.10	45.85	47.45	45.20	36.20	46.00

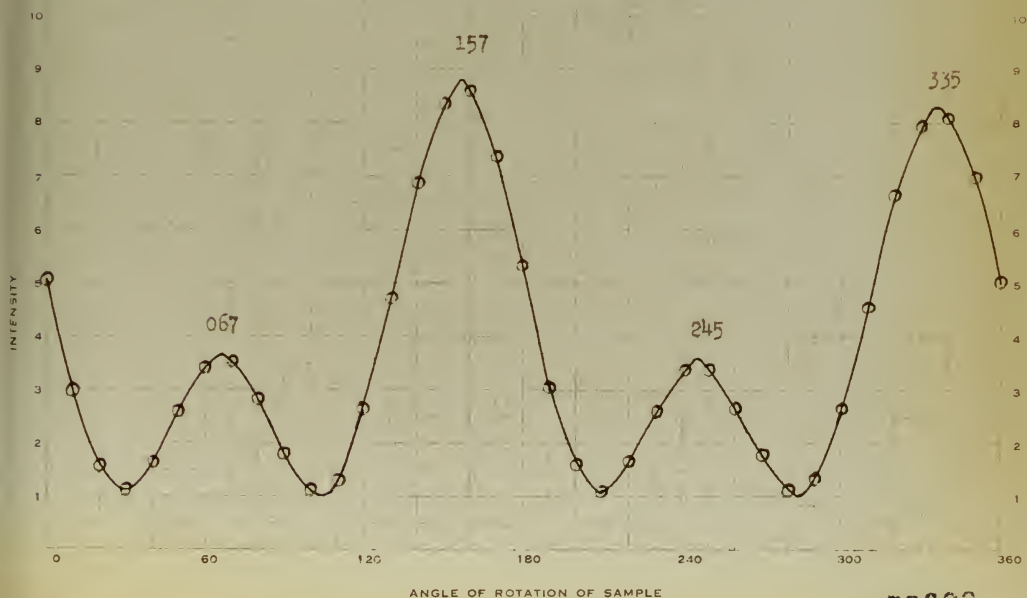
M	47.00	R	45.00
M	36.10	R	45.85
$\Delta M$	11.80	$\Delta R$	-.85

M <sub>1</sub>	47.45	R	45.20
M <sub>2</sub>	36.20	R	46.00
$\Delta M_{21}$	11.25	$\Delta R_{21}$	-.80

AV  $\Delta M$  11.53 PHASE SHIFTAV  $\Delta R$  -.83 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 10

115 FPM  $N_2$ 

DATE 2 APRIL 1952 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

12th. AUTOCLAVE CP 15 MIN. @ 21°C

@ 277		@ 097		@ 097		@ 127	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
42.0	45.0	36.7	45.7	47.3	45.0	36.8	45.8
48.0	45.1	36.5	45.8	47.5	45.0	36.2	45.8
48.00	45.05	36.50	45.75	47.40	45.00	36.50	45.80

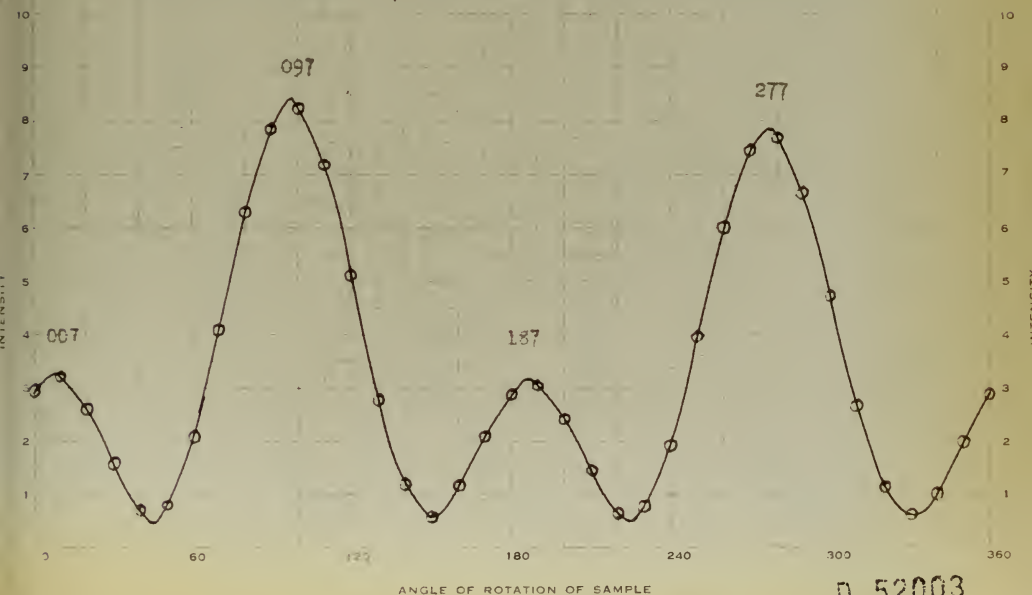
M <sub>1</sub>	48.00	R <sub>1</sub>	45.05
M <sub>2</sub>	36.50	R <sub>2</sub>	45.75
$\Delta M_{12}$	11.50	$\Delta R_{12}$	-.70

M <sub>3</sub>	47.40	R <sub>3</sub>	45.00
M <sub>4</sub>	36.50	R <sub>4</sub>	45.80
$\Delta M_{34}$	10.90	$\Delta R_{34}$	-.80

Av  $\Delta M$  11.15 PHASE SHIFTAv  $\Delta R$  -.75 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE No 13

113 PPM  $\lambda_2$ 

DATE 3 APRIL 1952 PM

CRYSTAL No 1

SURFACE FILM CONDITION

13th AUTOGRAVE OF 15 MIN. @ 233°C

@ 057		@ 147		@ 237		@ 327	
M <sub>1</sub>	R <sub>1</sub>	M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>1</sub>	M <sub>4</sub>	R <sub>4</sub>
47.8	44.8	36.4	46.0	47.9	44.8	36.2	45.7
47.9	44.8	36.2	45.8	47.7	44.8	36.3	45.9
AV 45.85	44.80	36.30	45.90	47.80	44.80	36.25	45.80

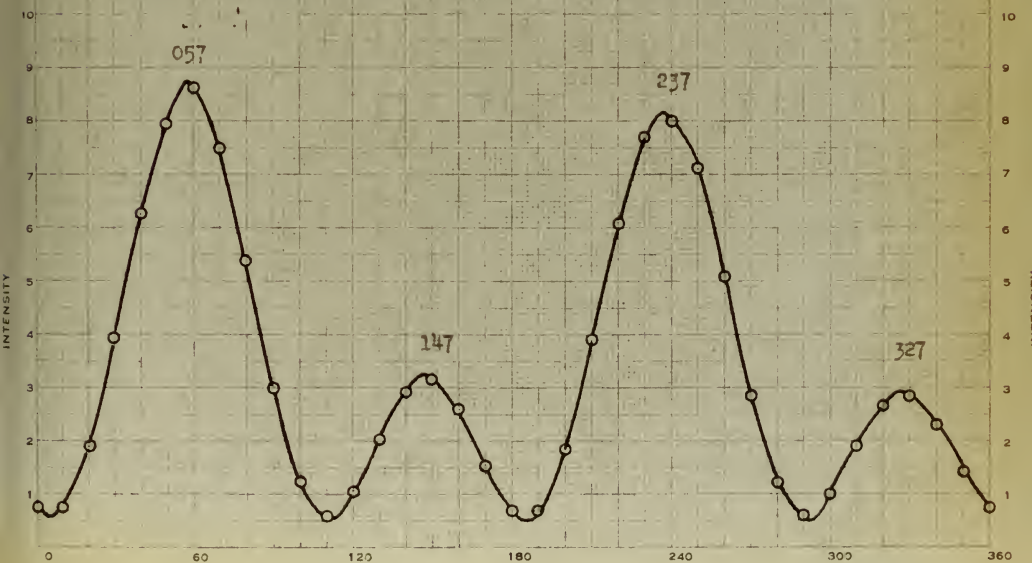
M <sub>1</sub>	47.85	R <sub>1</sub>	44.80
M <sub>2</sub>	36.30	R <sub>2</sub>	45.90
$\Delta M_{12}$	11.55	$\Delta R_{12}$	-1.10

M <sub>3</sub>	47.80	R <sub>3</sub>	44.80
M <sub>4</sub>	36.25	R <sub>4</sub>	45.80
$\Delta M_{34}$	11.55	$\Delta R_{34}$	-1.00

AV  $\Delta M$  11.55 PHASE SHIFTAV  $\Delta R$  -1.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

115

D 52003





SAMPLE NO 10

113 PPM N<sub>2</sub>

DATE 5 APRIL 1952 PM

CRYSTAL NO 1

SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 258		@ 088		@ 178		@ 268	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
49.6	45.1	38.4	46.4	49.7	45.2	38.0	46.2
50.0	45.1	38.2	46.3	49.7	45.3	37.6	46.4
49.80	45.10	38.30	46.35	49.7	45.25	37.80	46.30

M 49.80 R 45.10  
M<sub>2</sub> 38.30 R<sub>2</sub> 46.35  
ΔM<sub>12</sub> 11.50 ΔR<sub>12</sub> -1.25

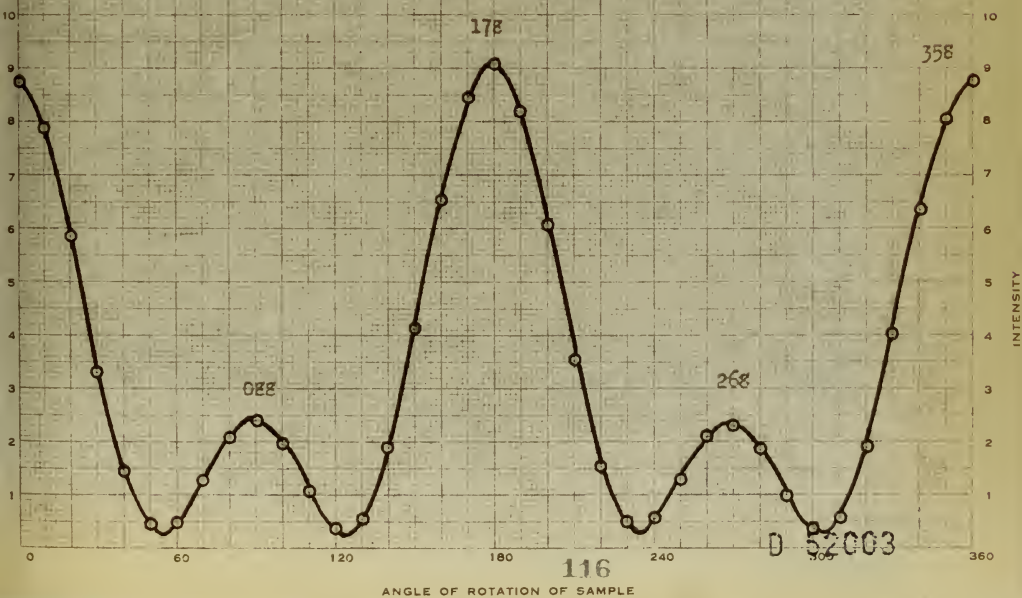
M<sub>3</sub> 49.70 R<sub>3</sub> 45.25  
M<sub>4</sub> 37.80 R<sub>4</sub> 46.30  
ΔM<sub>34</sub> 11.90 ΔR<sub>34</sub> -1.05

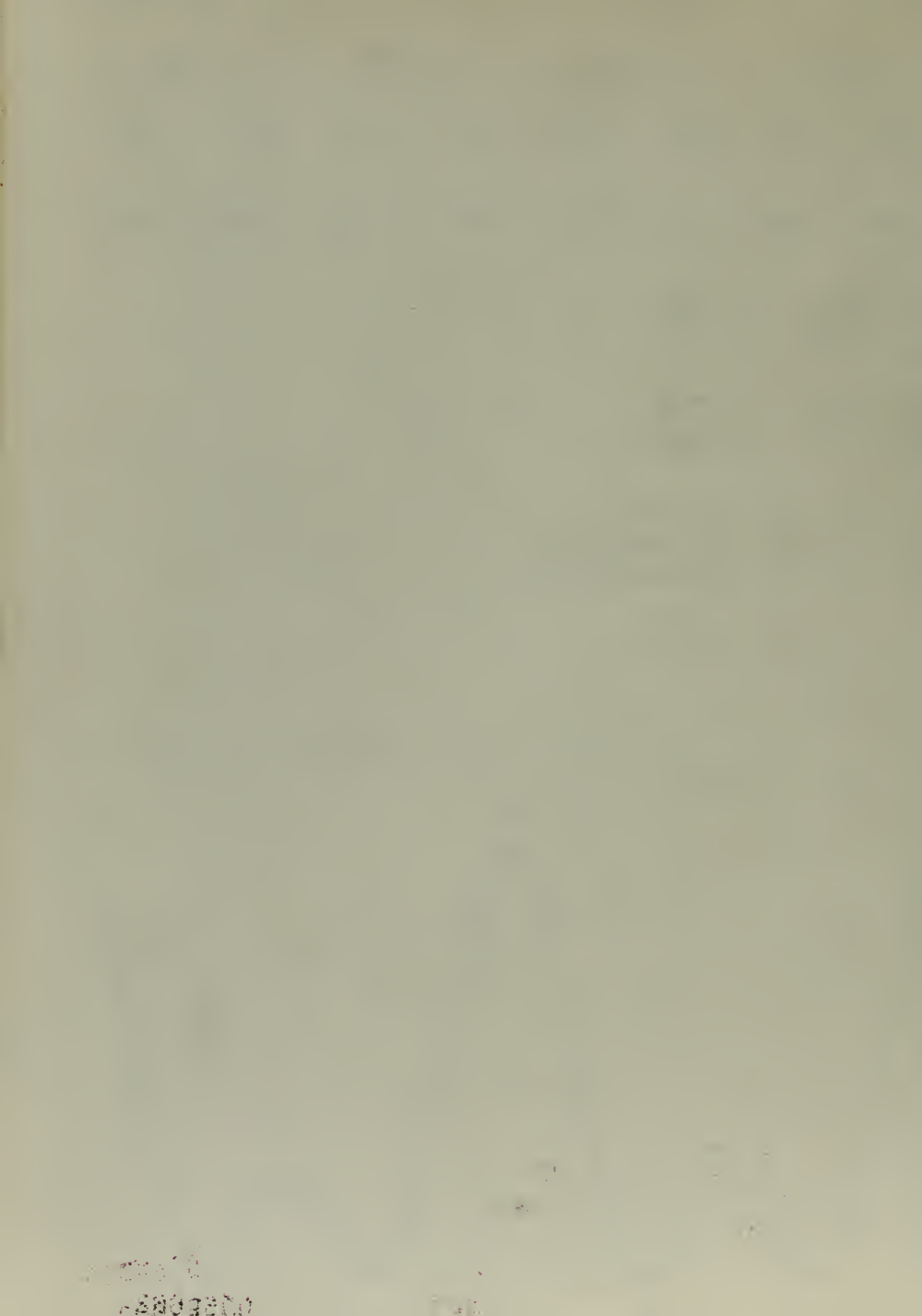
AV ΔM 11.70 PHASE SHIFT

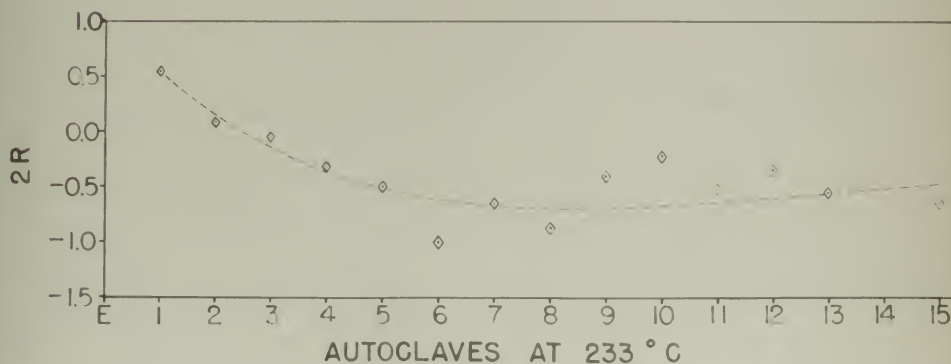
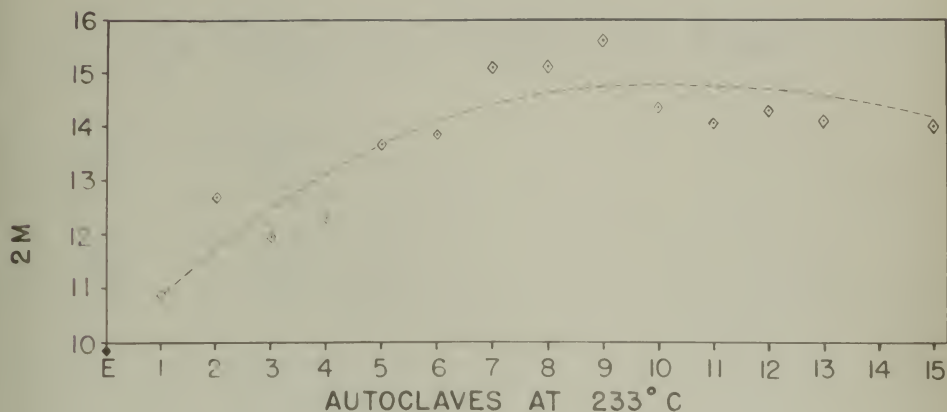
AV ΔR -1.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







PHASE SHIFT (2M) AND ROTATION OF PLANE OF  
POLARIZATION (2R) VS. CORROSION TIME  
FOR SAMPLE NO. ID-1



CRYSTAL No 1 SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. at 233°C

@ 039		@ 309		@ 219		@ 129	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.2	45.7	37.3	45.2	48.1	45.8	37.3	45.1
48.1	45.7	37.5	45.3	48.2	45.7	37.2	45.1
48.15	45.70	37.30	45.25	48.15	45.75	37.25	45.10

M <sub>1</sub>	48.15	R <sub>1</sub>	45.70
M <sub>2</sub>	37.30	R <sub>2</sub>	45.25
ΔM <sub>12</sub>	10.85	ΔR <sub>12</sub>	0.45

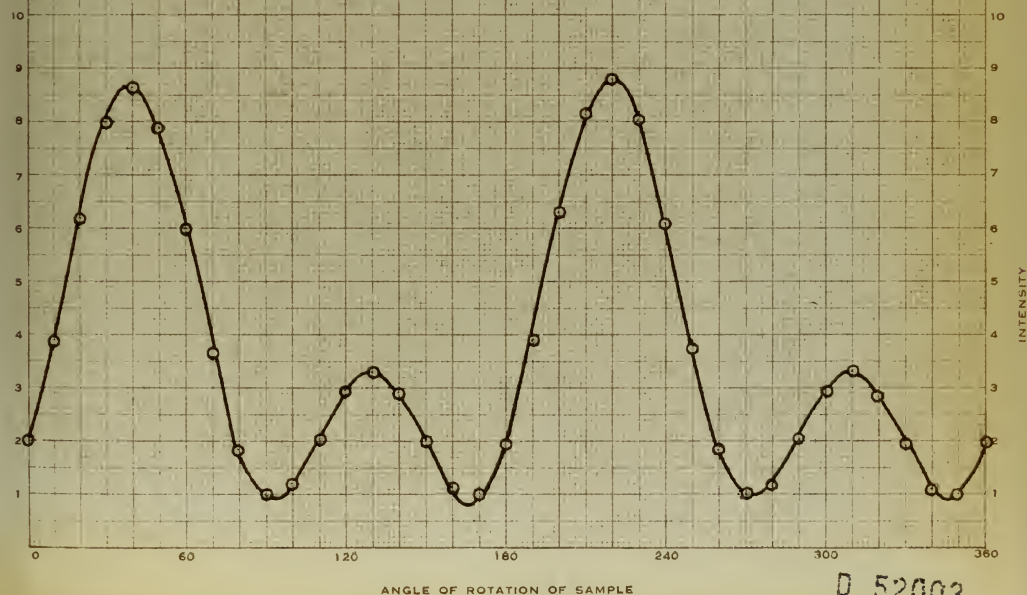
M <sub>3</sub>	48.15	R <sub>3</sub>	45.75
M <sub>4</sub>	37.25	R <sub>4</sub>	45.10
ΔM <sub>34</sub>	10.90	ΔR <sub>34</sub>	0.65

AV ΔM 10.87 PHASE SHIFT

AV ΔR 0.55 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE





SAMPLE NO. 1D

147 PPM N<sub>2</sub>

DATE 14 MARCH 1952 AM

CRYSTAL No 1

SURFACE FILM CONDITION

2nd AUTOCLAVE 15 MIN. at 233°C

AV

@ 294		@ 204		@ 114		@ 624	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
49.2	45.7	36.0	45.5	49.6	45.3	36.7	45.3
49.5	45.7	36.8	45.5	49.5	45.3	36.6	45.4
49.35	45.70	36.85	45.50	49.55	45.30	36.65	45.35

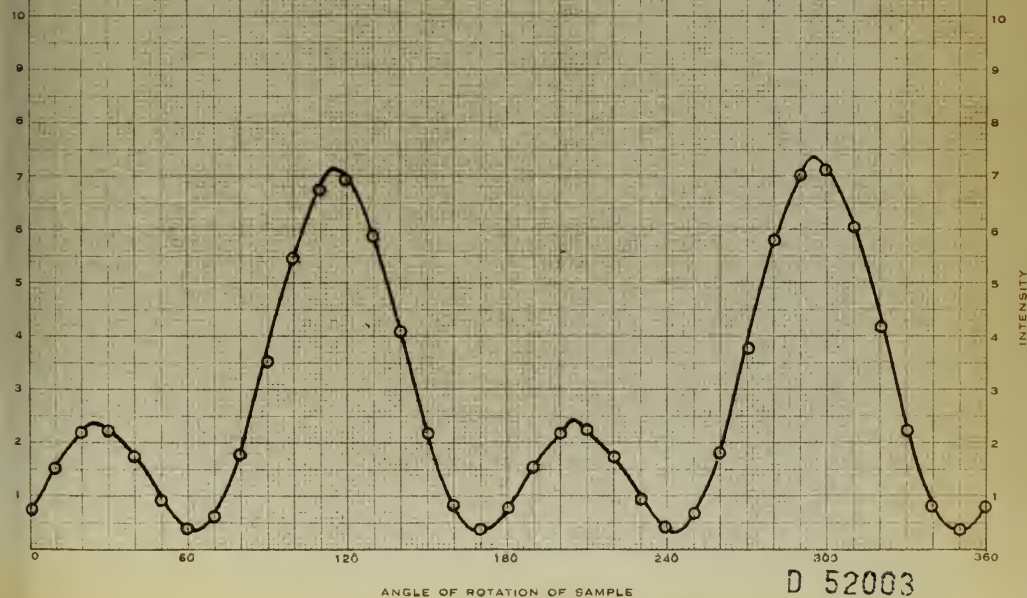
M <sub>1</sub>	49.35	R <sub>1</sub>	45.70
M <sub>2</sub>	36.85	R <sub>2</sub>	45.50
$\Delta M_{12}$	12.50	$\Delta R_{12}$	0.20

M <sub>3</sub>	49.55	R <sub>3</sub>	45.30
M <sub>4</sub>	36.65	R <sub>4</sub>	45.35
$\Delta M_{34}$	12.90	$\Delta R_{34}$	- .05

AV  $\Delta M$  12.70 PHASE SHIFTAV  $\Delta R$  .08 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 1D

147 PPM  $N_2$ 

DATE 17 MARCH 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION 3rd AUTOCLAVE 15 MIN. at 233°C

	@ 305		@ 215		@ 125		@ 035	
	M1	R1	M2	R2	M3	R3	M4	R4
	48.7	45.5	36.4	45.7	48.7	45.7	36.9	45.5
	48.5	45.6	36.5	45.6	48.2	45.5	36.5	45.5
					48.5	45.6	36.8	45.5
AV	48.60	45.55	36.45	45.65	48.47	45.60	36.73	45.60

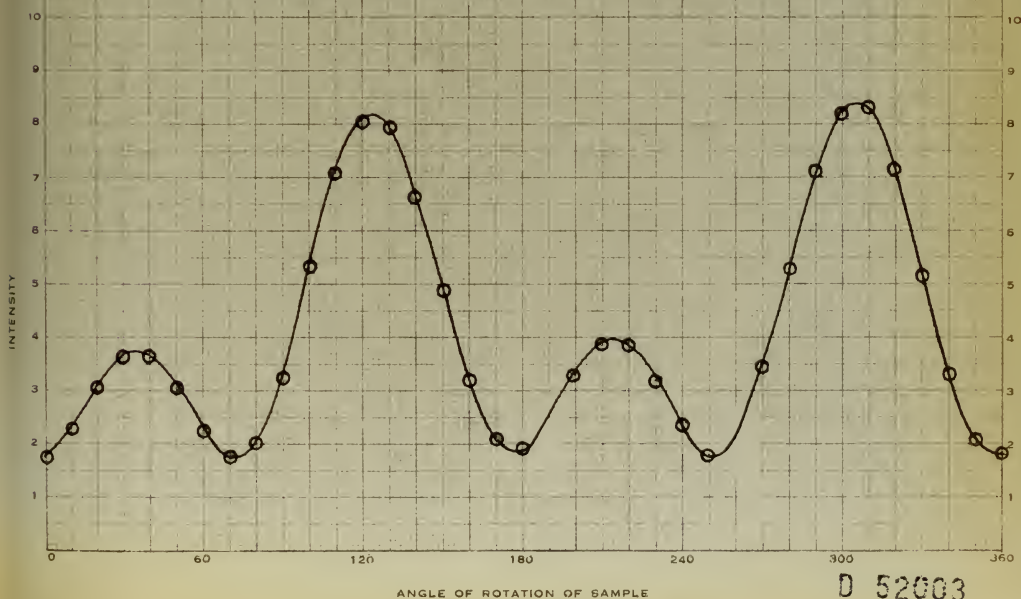
M <sub>1</sub>	48.60	R <sub>1</sub>	45.55
M <sub>2</sub>	36.45	R <sub>2</sub>	45.65
$\Delta M_{12}$	12.15	$\Delta R_{12}$	- .10

M <sub>3</sub>	48.47	R <sub>3</sub>	45.60
M <sub>4</sub>	36.73	R <sub>4</sub>	45.60
$\Delta M_{34}$	11.74	$\Delta R_{34}$	0.00

AV  $\Delta M$  11.95 PHASE SHIFTAV  $\Delta R$  -0.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE No 1D

147 PPM  $V_2$ 

DATE 22 MARCH 1952 AM

CRYSTAL No 1

SURFACE FILM COMPOSITION

4th AUTOCLAVE 15 MIN. @ 233°C

@ 013		@ 283		@ 193		@ 103	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.8	45.3	35.7	45.7	47.9	45.3	35.7	45.6
48.2	45.3	35.5	45.8	47.7	45.5	35.5	45.6
AV 48.00	45.30	35.60	45.75	47.80	45.40	35.60	45.60

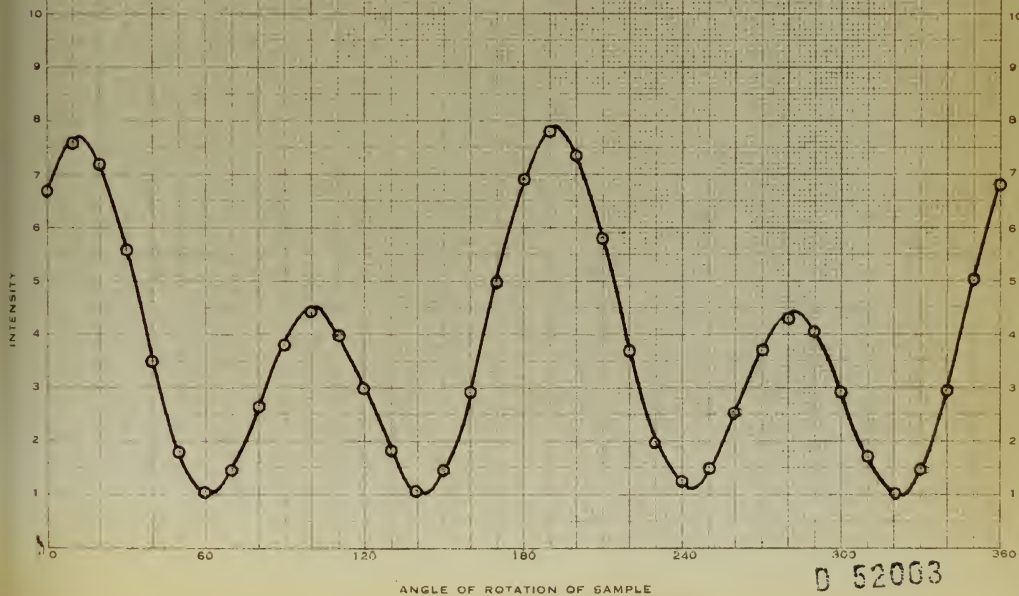
M <sub>1</sub> 48.00	R <sub>1</sub> 45.30
M <sub>2</sub> 35.60	R <sub>2</sub> 45.75
$\Delta M_{12}$ 12.40	$\Delta R_{12}$ -0.45

M <sub>3</sub> 47.80	R <sub>3</sub> 45.40
M <sub>4</sub> 35.60	R <sub>4</sub> 45.60
$\Delta M_{34}$ 12.20	$\Delta R_{34}$ -0.20

AV  $\Delta M$  12.30 PHASE SHIFTAV  $\Delta R$  -0.32 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE No 10

147 PPM  $H_2$ 

DATE 24 MARCH 1952 PM

CRYSTAL No 1

SURFACE FILM CONDITION

564 APR 02 72 15 MIN. © 2370

@ 153		@ 263		@ 173		@ 083	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.9	45.3	34.2	45.8	47.9	45.4	34.2	45.8
47.8	45.3	34.3	45.7	47.9	45.3	34.1	45.8
AV 47.85	45.30	34.25	45.75	47.90	45.35	34.15	45.80

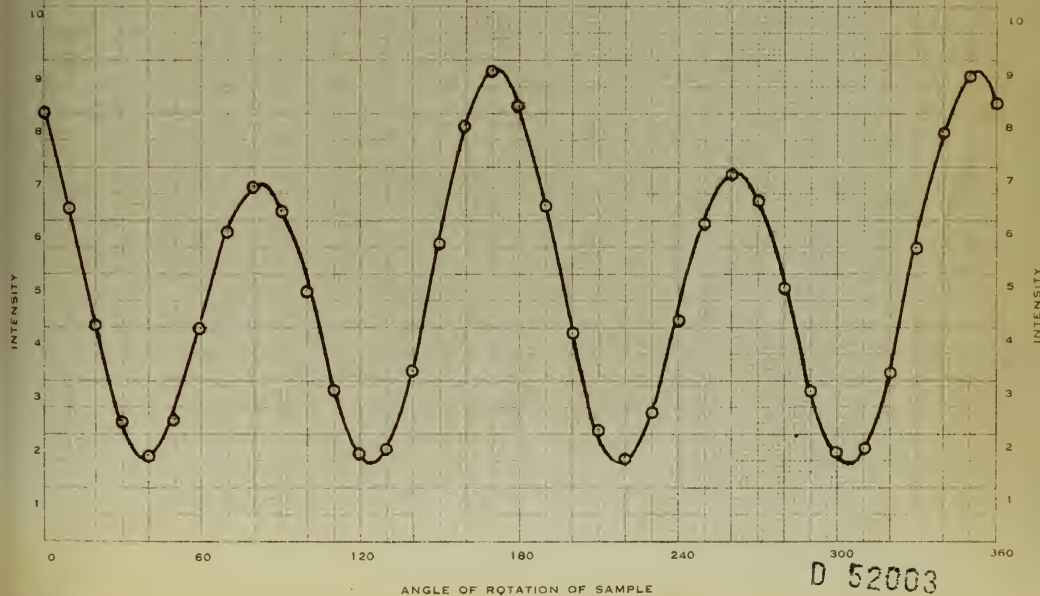
M<sub>1</sub> 47.85 R<sub>1</sub> 45.30  
M<sub>2</sub> 34.25 R<sub>2</sub> 45.75  
 $\Delta M_{12}$  13.60  $\Delta R_{12}$  -0.45

M<sub>3</sub> 47.90 R<sub>3</sub> 45.35  
M<sub>4</sub> 34.15 R<sub>4</sub> 45.80  
 $\Delta M_{34}$  13.75  $\Delta R_{34}$  -0.55

AV  $\Delta M$  13.67 PHASE SHIFTAV  $\Delta R$  -0.50 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE NO 1D

147PPM  $N_2$ 

DATE 25 MARCH 1958 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

6th AUTOCLAVE 15 MIN. @ 233°C

@ 346		@ 356		@ 366		@ 376	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.3	45.2	36.6	46.3	50.3	45.5	36.4	46.5
50.3	45.2	36.5	46.2	50.4	45.6	36.4	46.5
AV 50.30	45.20	36.55	46.25	50.35	45.55	36.40	46.50

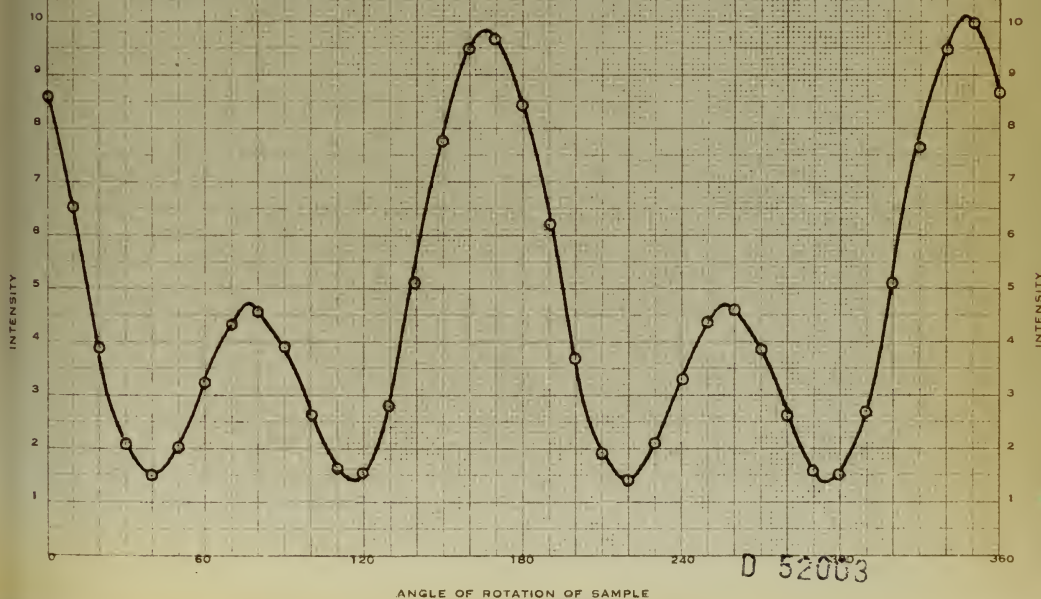
M <sub>1</sub>	50.30	R <sub>1</sub>	45.20
M <sub>2</sub>	36.55	R <sub>2</sub>	46.25
$\Delta M_{12}$	13.75	$\Delta R_{12}$	-1.05

M <sub>3</sub>	50.35	R <sub>3</sub>	45.55
M <sub>4</sub>	36.40	R <sub>4</sub>	46.50
$\Delta M_{34}$	13.95	$\Delta R_{34}$	-0.95

AV  $\Delta M$  13.85 PHASE SHIFTAV  $\Delta R$  -1.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE



SAMPLE No. 1D

147 PPM N<sub>2</sub>

DATE 26 MARCH 1952 PM

CRYSTAL No. 1

SURFACE FILM CONDITION

7th AUTOCLAVE 15 MIN. @ 233°C

@ 331

@ 241

@ 151

@ 061

M<sub>1</sub>R<sub>1</sub>M<sub>2</sub>R<sub>2</sub>M<sub>3</sub>R<sub>3</sub>M<sub>4</sub>R<sub>4</sub>

50.5

45.4

35.3

46.0

50.2

45.4

35.2

46.1

50.6

45.3

35.3

46.1

50.3

45.5

35.4

46.0

AV.

50.55

45.35

35.30

46.05

50.25

45.45

35.30

46.05

M<sub>1</sub> 50.55R<sub>1</sub> 45.35M<sub>2</sub> 35.30R<sub>2</sub> 46.05 $\Delta M_{12}$  15.25 $\Delta R_{12}$  -0.70M<sub>3</sub> 50.25R<sub>3</sub> 45.45M<sub>4</sub> 35.30R<sub>4</sub> 46.05 $\Delta M_{34}$  14.95 $\Delta R_{34}$  -0.60AV  $\Delta M$  15.10

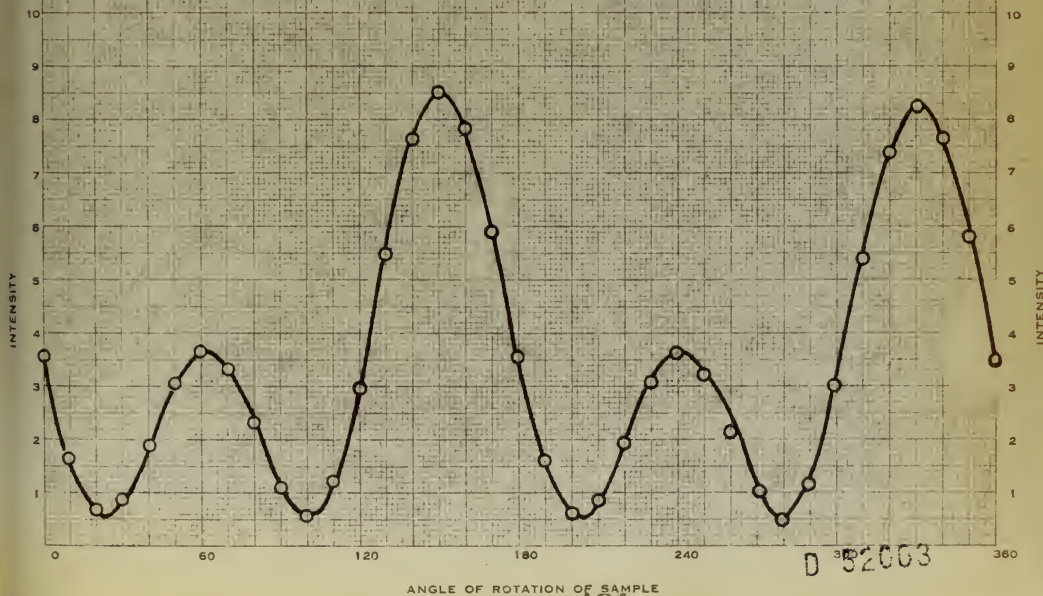
PHASE SHIFT

AV  $\Delta R$  -0.65

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 82003





SAMPLE No 12

147 PPM H<sub>2</sub>

DATE 27 MARCH 1951 PM

CRYSTAL No 1

SURFACE FILM CONDITION

8th AUTOCLAVE 15 MIN. @ 233°C

@ 005		@ 276		@ 186		@ 095	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.2	45.0	37.1	46.1	51.6	45.1	36.5	45.8
52.2	45.1	36.9	45.9	51.6	45.1	36.5	46.0
Av 52.20	45.05	37.00	46.00	51.60	45.10	36.55	45.90

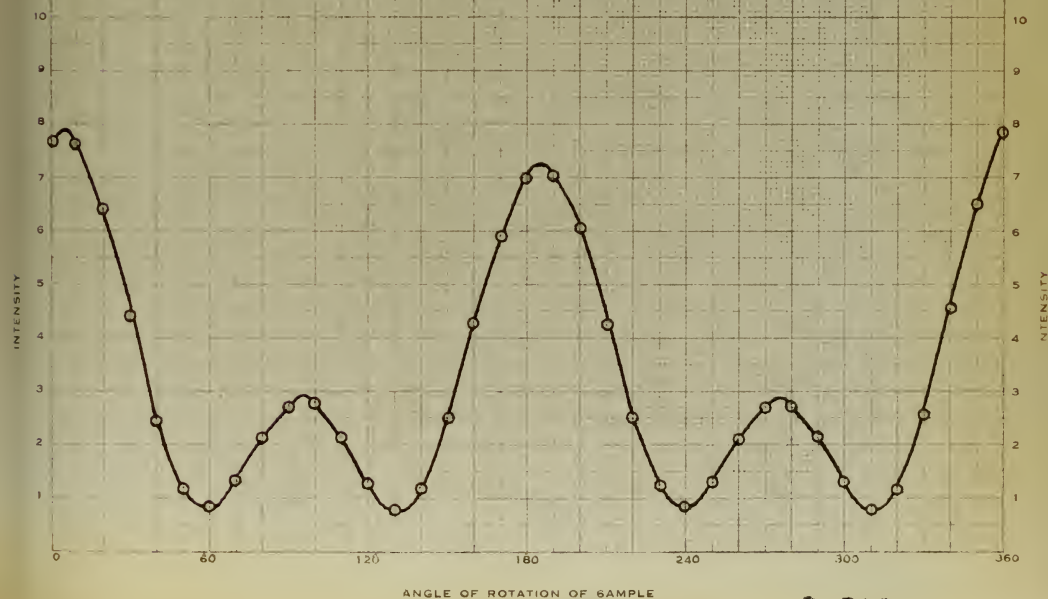
M<sub>1</sub> 52.20      R<sub>1</sub> 45.05  
 M<sub>2</sub> 37.00      R<sub>2</sub> 46.00  
 $\Delta M_{12}$  15.20     $\Delta R_{12}$  -0.95

M<sub>3</sub> 51.60      R<sub>3</sub> 45.10  
 M<sub>4</sub> 36.55      R<sub>4</sub> 45.90  
 $\Delta M_{34}$  15.05     $\Delta R_{34}$  -0.80

Av  $\Delta M$  15.12      PHASE SHIFTAv  $\Delta R$  -0.37      PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE No 1D

147 PPM N<sub>2</sub>

DATE 29 MARCH 1952 AM

CRYSTAL No 1

SURFACE FILM CONDITION

9th AUTOCLAVE 15 MIN @ 233°C

@ 116		@ 226		@ 136		@ 046	
M	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.4	45.6	36.7	46.0	51.9	45.7	36.4	46.0
52.5	45.5	37.0	46.1	51.9	45.7	36.2	46.0
Av 52.45	45.55	36.85	46.05	51.90	45.70	36.30	46.00

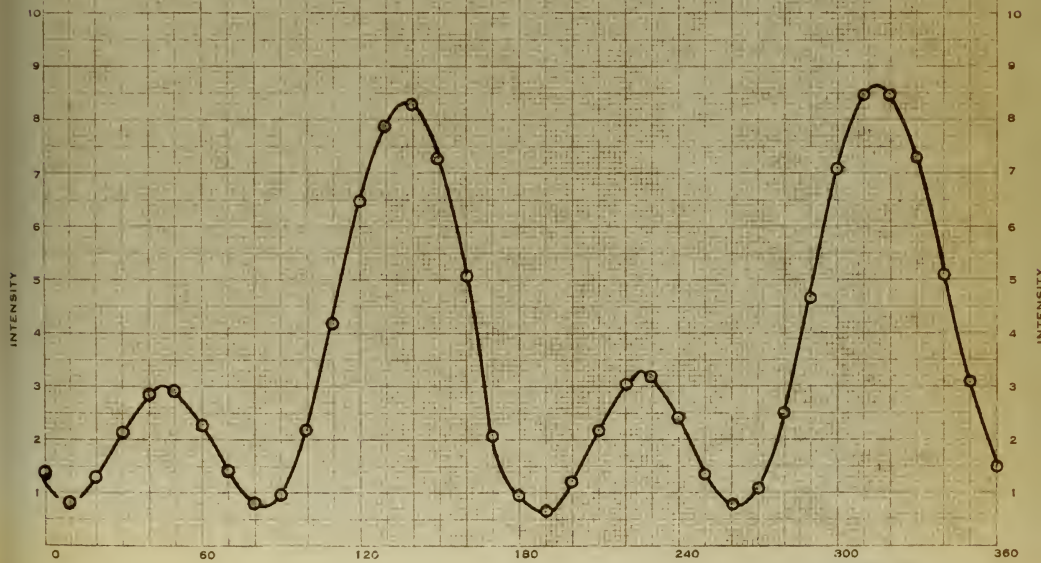
M <sub>1</sub>	52.45	R <sub>1</sub>	45.55
M <sub>2</sub>	36.85	R <sub>2</sub>	46.05
$\Delta M_{12}$	15.60	$\Delta R_{12}$	-0.50

M <sub>3</sub>	51.90	R <sub>3</sub>	45.70
M <sub>4</sub>	36.30	R <sub>4</sub>	46.00
$\Delta M_{34}$	15.60	$\Delta R_{34}$	-0.30

Av  $\Delta M$  15.60 PHASE SHIFTAv  $\Delta R$  -0.40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 10

147 PPM  $\lambda_2$ 

DATE 31 MARCH 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION

1<sup>st</sup> AUTOCLAVE 15 MIN. @ 233°C

@ 337	@ 347	@ 357	@ 367
M <sub>1</sub>	M <sub>2</sub>	M <sub>3</sub>	M <sub>4</sub>
50.6	45.8	36.5	46.1
50.3	45.7	36.2	46.0
AV: 50.45	45.75	36.35	46.05

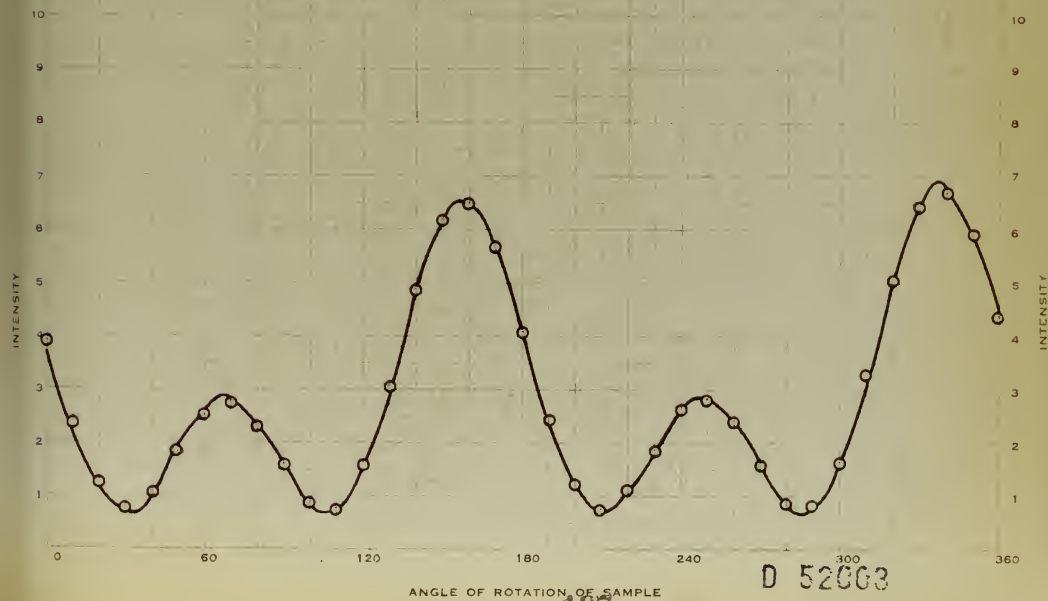
M <sub>1</sub>	50.45	R <sub>1</sub>	45.75
M <sub>2</sub>	36.35	R <sub>2</sub>	46.05
$\Delta M_2$	14.10	$\Delta R_2$	-.30

M <sub>3</sub>	50.50	R <sub>3</sub>	45.35
M <sub>4</sub>	35.85	R <sub>4</sub>	45.70
$\Delta M_{34}$	14.65	$\Delta R_{34}$	-.15

AV  $\Delta M$  14.37 PHASE SHIFTAV  $\Delta R$  -.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 1D

147 PPM  $N_2$ 

DATE 1 APRIL 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION

11th AUTOCLAVE 15 MIN. @ 233°C

@ 274		@ 184		@ 094		@ 004	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.4	45.1	36.0	45.6	49.7	45.2	35.7	45.9
50.1	45.1	36.3	45.6	49.8	45.3	35.7	45.7
AV. 50.25	45.10	36.15	45.60	49.75	45.25	35.70	45.80

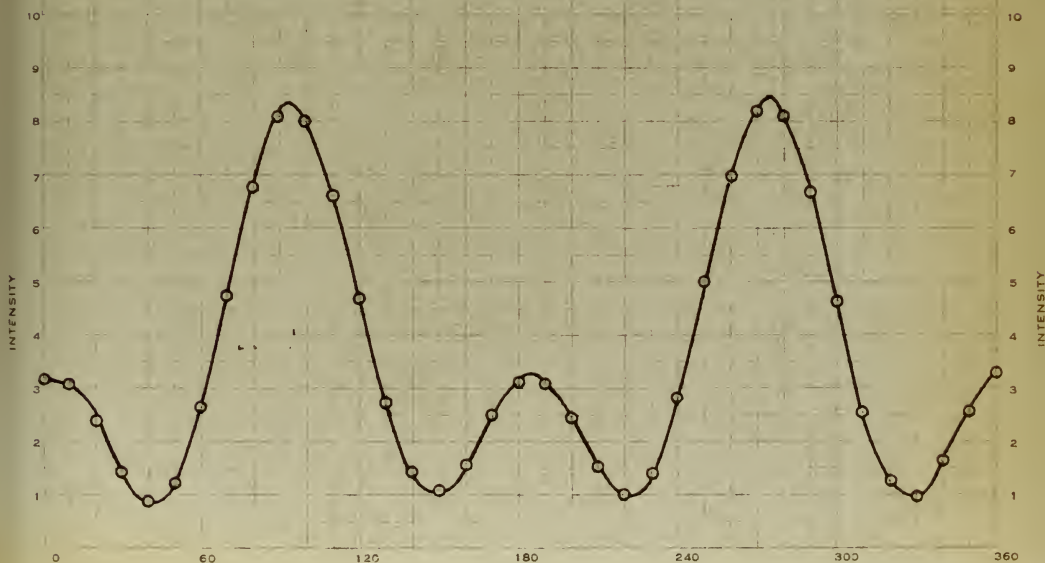
M <sub>1</sub>	50.25	R <sub>1</sub>	45.10
M <sub>2</sub>	36.15	R <sub>2</sub>	45.60
$\Delta M_{12}$	14.10	$\Delta R_{12}$	-.50

M <sub>3</sub>	49.75	R <sub>3</sub>	45.25
M <sub>4</sub>	35.70	R <sub>4</sub>	45.80
$\Delta M_{34}$	14.05	$\Delta R_{34}$	-.55

AV  $\Delta M$  14.07 PHASE SHIFTAV  $\Delta R$  -.52 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







CRYSTAL NO. 1 SURFACE FILM CONDITION 12th AUTOCLAVE 15 MIN. @ 233°C

@ 203		@ 113		@ 023		@ 293	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
49.1	45.3	35.0	45.8	49.5	45.2	34.1	45.5
49.3	45.3	35.3	45.7	49.3	45.4	35.0	45.6
AV 49.20	45.30	35.15	45.75	49.40	45.30	34.85	45.55

M	49.20	R	45.30
M <sub>1</sub>	35.15	R	45.75
$\Delta M_{12}$	14.05	$\Delta R_{12}$	-0.45

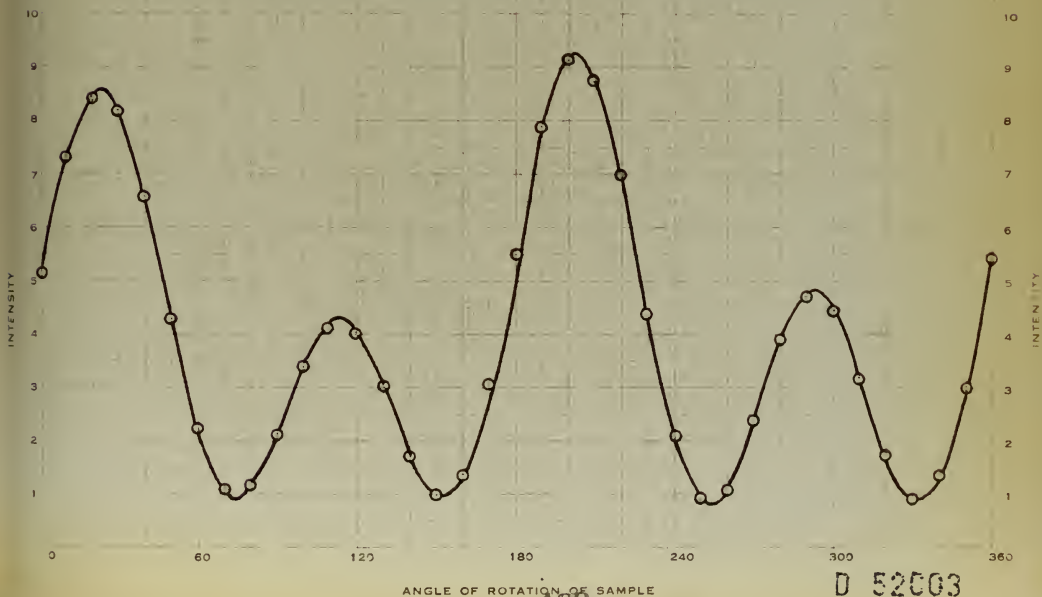
M	49.40	R <sub>3</sub>	45.30
M <sub>4</sub>	34.85	R <sub>4</sub>	45.55
$\Delta M_{34}$	14.55	$\Delta R_{34}$	-0.25

AV  $\Delta M$  14.30 PHASE SHIFT

AV  $\Delta R$  -0.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO. 1D

147 PPM  $N_2$ 

DATE 4 APRIL 1952 AM

CRYSTAL NO. 1

SURFACE FILM, CONDITION.

13th AUTOCLAVE 15 MIN. @ 233°C

@ 216		@ 126		@ 036		@ 306	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
49.8	45.4	35.4	45.8	49.2	45.8	35.0	46.4
49.1	45.8	35.4	46.3	49.2	45.5	35.3	46.2
49.5	45.5						
AV 49.57	45.57	35.40	46.05	49.20	45.65	35.15	46.30

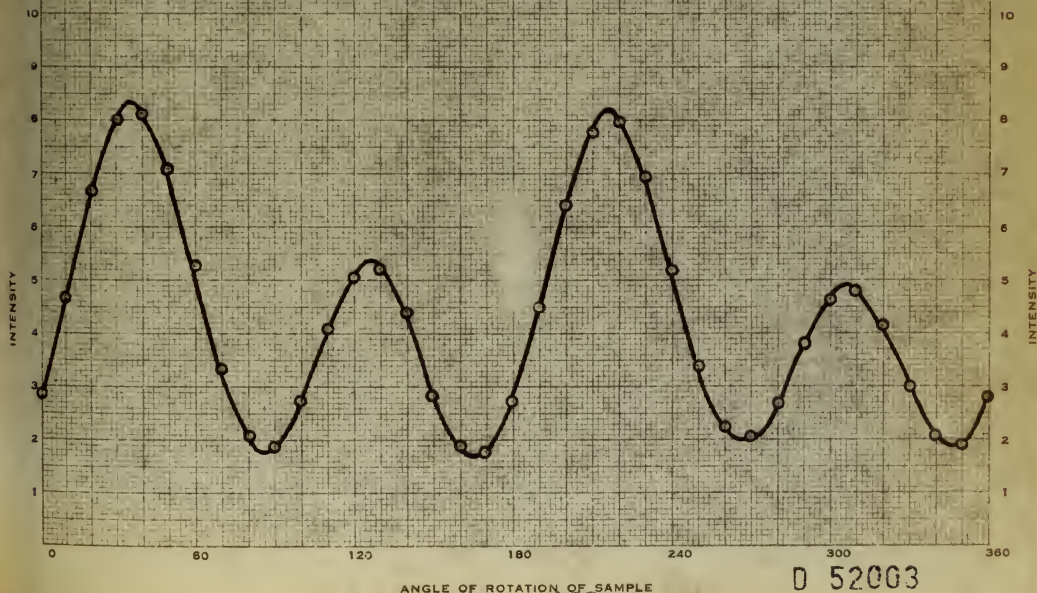
$M_1$  49.57       $R_1$  45.57  
 $M_2$  35.40       $R_2$  46.05  
 $\Delta M_{12}$  14.17       $\Delta R_{12}$  -.48

$M_3$  49.20       $R_3$  45.65  
 $M_4$  35.15       $R_4$  46.30  
 $\Delta M_{34}$  14.05       $\Delta R_{34}$  -.65

AV  $\Delta M$  14.11 PHASE SHIFTAV  $\Delta R$  -0.56 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE NO 1D

147 PPM  $N_2$ 

DATE 5 APRIL 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION 15th AUTOCLAVE of 15 MIN. @ 233°C

@ 336		@ 246		@ 156		@ 66	
M1	R1	M2	R2	M3	R3	M4	R4
51.4	45.7	37.7	46.50	51.1	45.50	36.8	46.1
51.2	45.7	37.5	46.40	51.0	45.70	36.6	46.2
AV 51.30		45.70		37.65		46.45	
51.05		45.60		36.70		46.15	

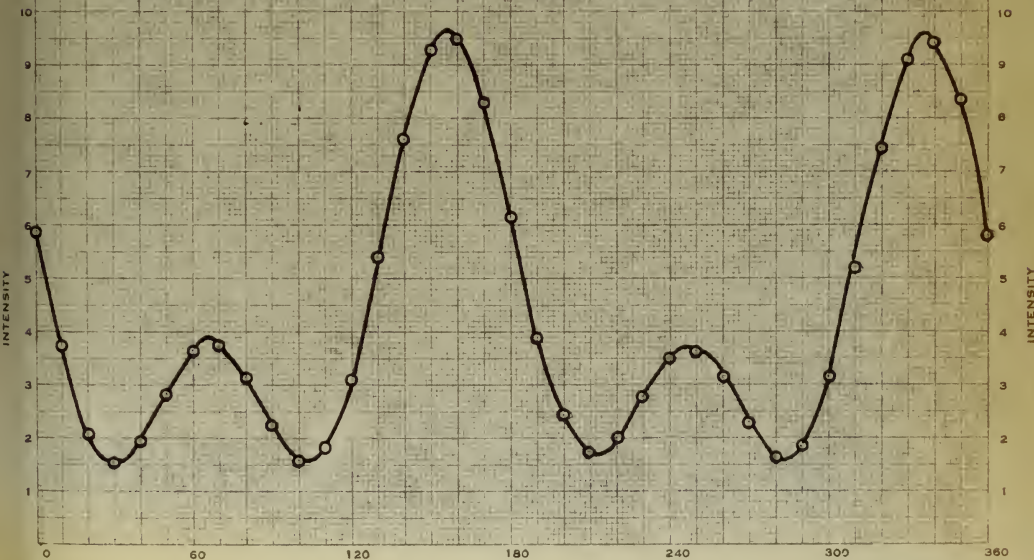
M <sub>1</sub>	51.30	R	45.70
M <sub>2</sub>	37.65	R <sub>2</sub>	46.45
$\Delta M_{12}$	13.65	$\Delta R_{12}$	- .75

M <sub>3</sub>	51.05	R <sub>3</sub>	45.60
M <sub>4</sub>	36.70	R <sub>4</sub>	46.15
$\Delta M_{34}$	14.35	$\Delta R_{34}$	- .55

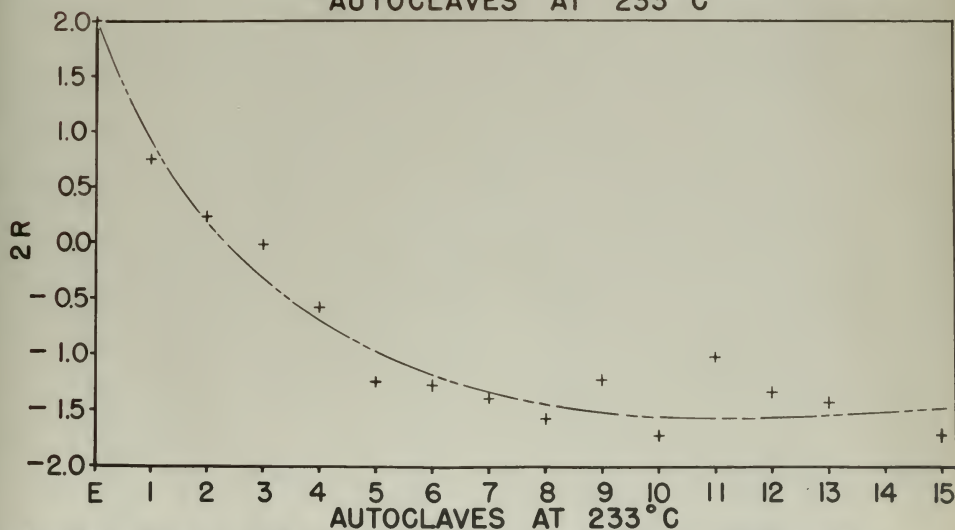
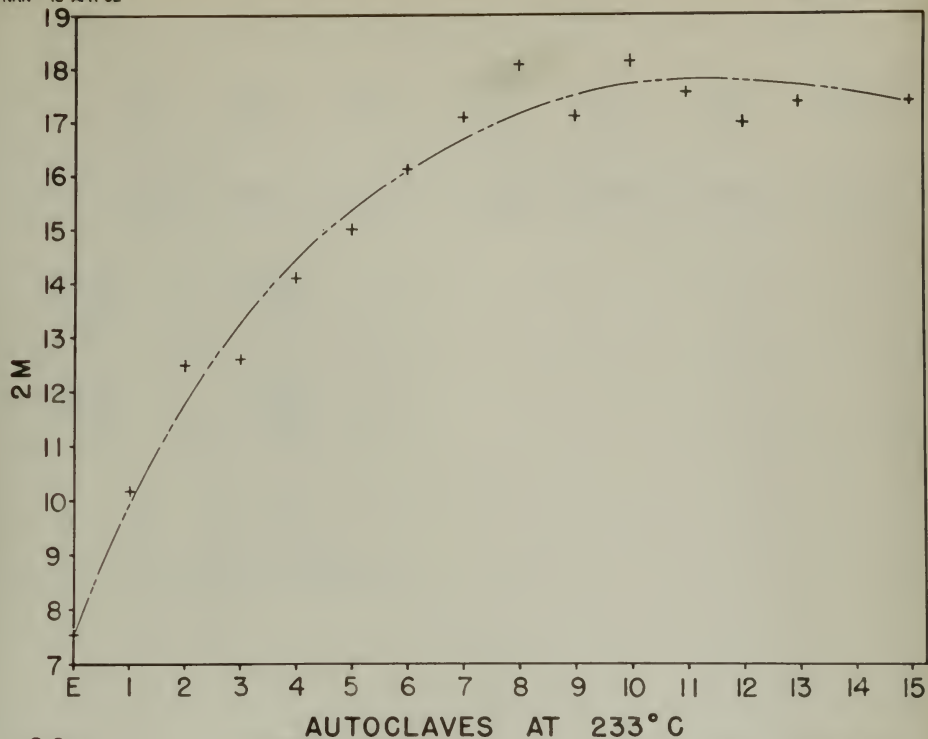
AV  $\Delta M$  14.00 PHASE SHIFTAV  $\Delta R$  -0.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







PHASE SHIFT ( $2m$ ) AND ROTATION OF PLANE OF  
POLARIZATION ( $2r$ ) VS. CORROSION TIME FOR  
SAMPLE NO. 1E - 1





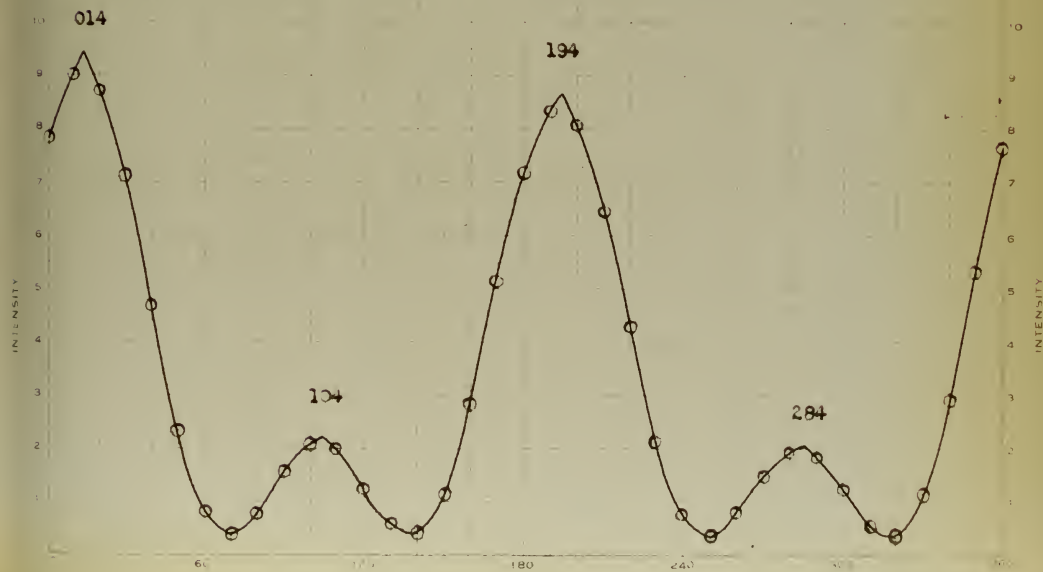
(014)		(0104)		(0194)		(0284)	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.1	46.4	39.0	44.2	46.4	46.5	39.3	44.6
46.9	46.5	39.4	44.4	46.8	46.3	39.3	44.5
47.00	46.45	39.20	44.30	46.60	46.40	39.30	44.55

M <sub>1</sub>	47.00	R <sub>1</sub>	46.45
M <sub>2</sub>	39.20	R <sub>2</sub>	44.30
ΔM <sub>1</sub>	7.80	ΔR <sub>1</sub>	2.15

M <sub>3</sub>	46.60	R <sub>3</sub>	46.40
M <sub>4</sub>	39.30	R <sub>4</sub>	44.55
ΔM <sub>3</sub>	7.30	ΔR <sub>3</sub>	1.85

Av ΔM 7.55 PHASE SHIFT  
Av ΔR 2.00 PHASE ROTATION  
PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CRYSTAL NO

SURFACE FILM CONDITION

①		②		③		④	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.8	50.0	50.8	48.2	41.0	41.1	50.5	48.0
48.8	50.0	50.8	48.2	41.0	41.1	50.5	48.0
AV	48.85	45.98	38.68	40.75	40.40	45.40	45.30

M <sub>1</sub>	48.85	R <sub>1</sub>	45.98
M <sub>2</sub>	50.8	R <sub>2</sub>	48.2
ΔM <sub>12</sub>	2.95	ΔR <sub>12</sub>	2.28

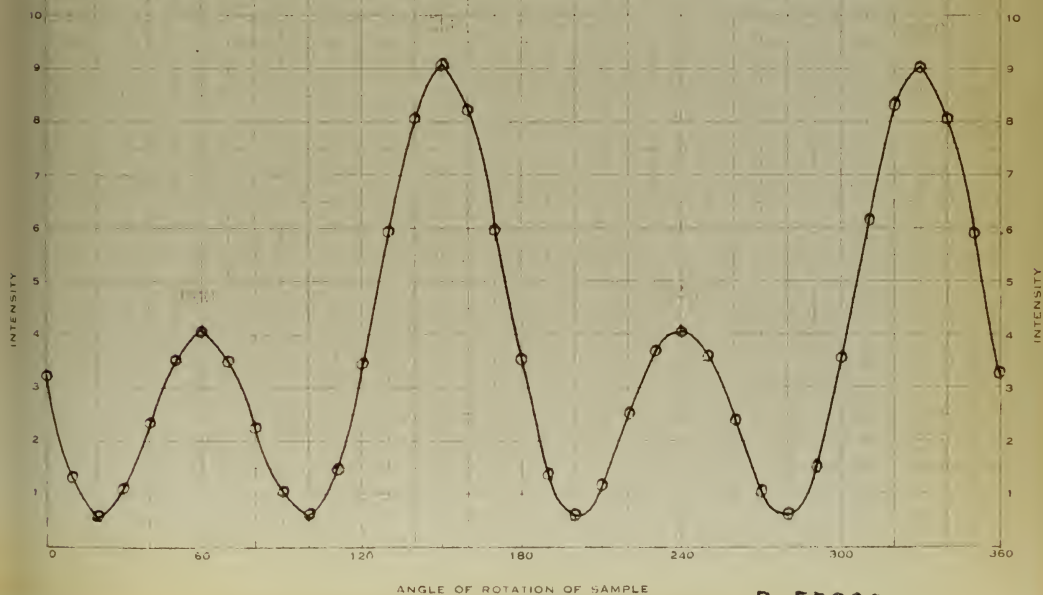
M <sub>3</sub>	41.0	R <sub>3</sub>	41.1
M <sub>4</sub>	50.5	R <sub>4</sub>	48.0
ΔM <sub>34</sub>	9.5	ΔR <sub>34</sub>	6.9

AV ΔM 17.75 PHASE SHIFT

AV ΔR 7.5 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CRYSTAL NO

SURFACE FILM CONDITION

@ 74°		@ 90°		@ 106°		@ 124°	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
41.2	46.5	50.5	41.1	49.2	48.0	36.2	41.2
41.4	46.7	41.5	45.4	49.2	48.2	36.2	45.6
Av	41.30	46.50	44.56	48.40	47.25	41.05	40.40

M <sub>1</sub>	41.2	R <sub>1</sub>	46.5
M <sub>2</sub>	41.4	R <sub>2</sub>	46.7
ΔM <sub>12</sub>	0.2	ΔR <sub>12</sub>	-0.2

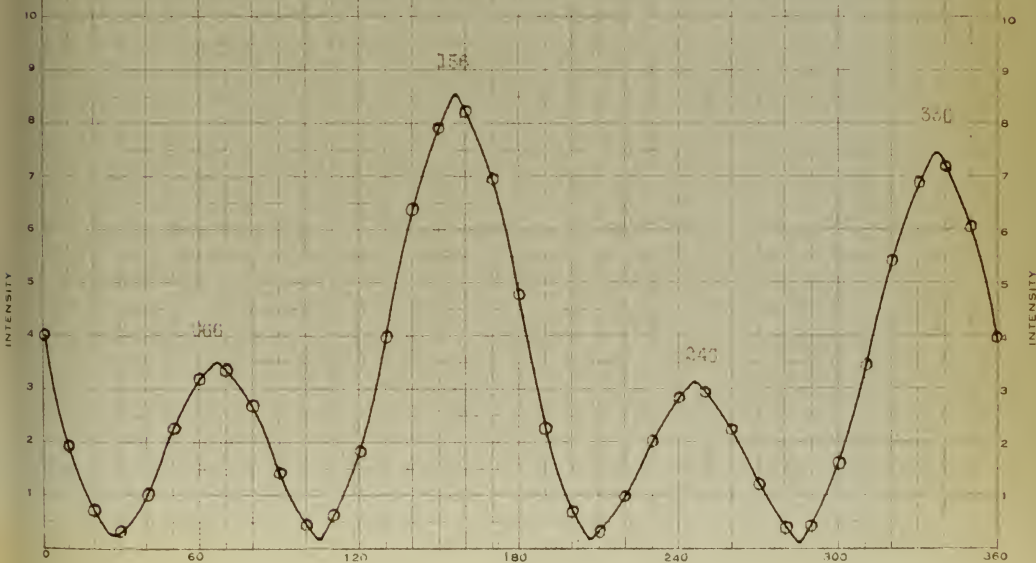
M <sub>3</sub>	49.2	R <sub>3</sub>	48.0
M <sub>4</sub>	36.2	R <sub>4</sub>	41.2
ΔM <sub>34</sub>	13.0	ΔR <sub>34</sub>	7.2

Av ΔM 10.40 PHASE SHIFT

Av ΔR 4.28 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







CRYSTAL No

SURFACE FILM CONDITION

①		②		③		④	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
41.1	40.4	38.1	45.5	40.2	45.4	38.2	45.5
41.0	45.5	38.1	45.5	40.2	45.4	38.2	45.5
Av	41.4	41.45	41.45	41.45	41.45	41.45	41.45

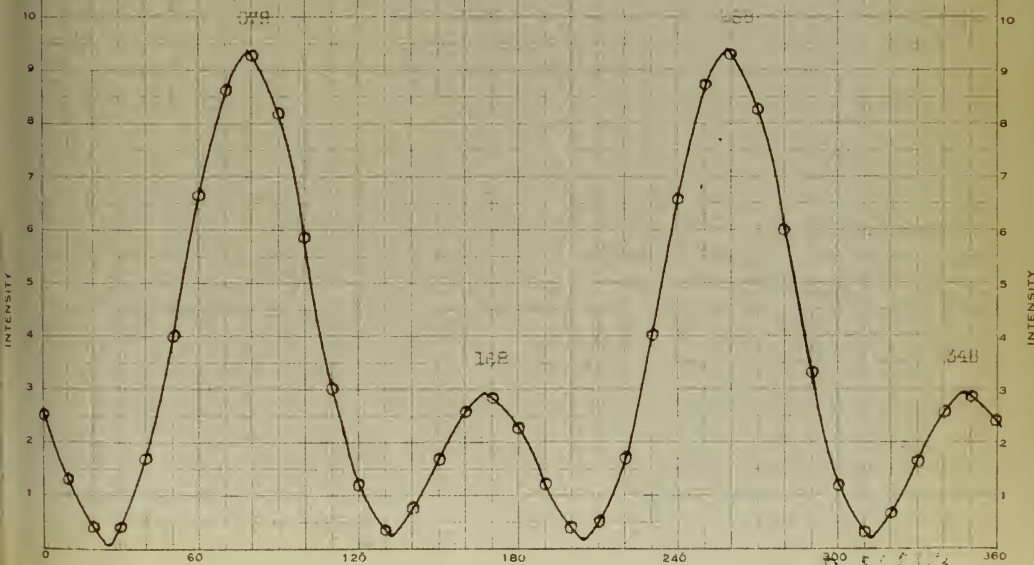
M <sub>1</sub>	45.05	R <sub>1</sub>	45.45
M <sub>2</sub>	45.50	R <sub>2</sub>	45.45
$\Delta M_{12}$	1.55	$\Delta R_{12}$	- .05

M <sub>3</sub>	45.45	R <sub>3</sub>	45.45
M <sub>4</sub>	45.45	R <sub>4</sub>	45.45
$\Delta M_{34}$	1.60	$\Delta R_{34}$	0

Av  $\Delta M$  12.54 PHASE SHIFTAv  $\Delta R$  - .1 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







CRYSTAL No 1 SURFACE FILM CONDITION 4th AUTOCLAVE OF 15 MIN. at 233°C

@ <u>005</u>		@ <u>095</u>		@ <u>185</u>		@ <u>275</u>	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.8	45.5	34.7	46.1	48.5	45.5	34.7	46.0
48.9	45.5	34.9	46.2	48.6	45.5	34.3	46.0
AV. 48.85	45.50	34.80	46.15	48.60	45.50	34.50	46.00

M <sub>1</sub>	48.85	R <sub>1</sub>	45.50
M <sub>2</sub>	34.80	R <sub>2</sub>	46.15
$\Delta M_{12}$	14.05	$\Delta R_{12}$	-.65

M <sub>3</sub>	48.50	R <sub>3</sub>	45.50
M <sub>4</sub>	34.50	R <sub>4</sub>	46.00
$\Delta M_{34}$	14.10	$\Delta R_{34}$	-.50

AV  $\Delta M$  14.08

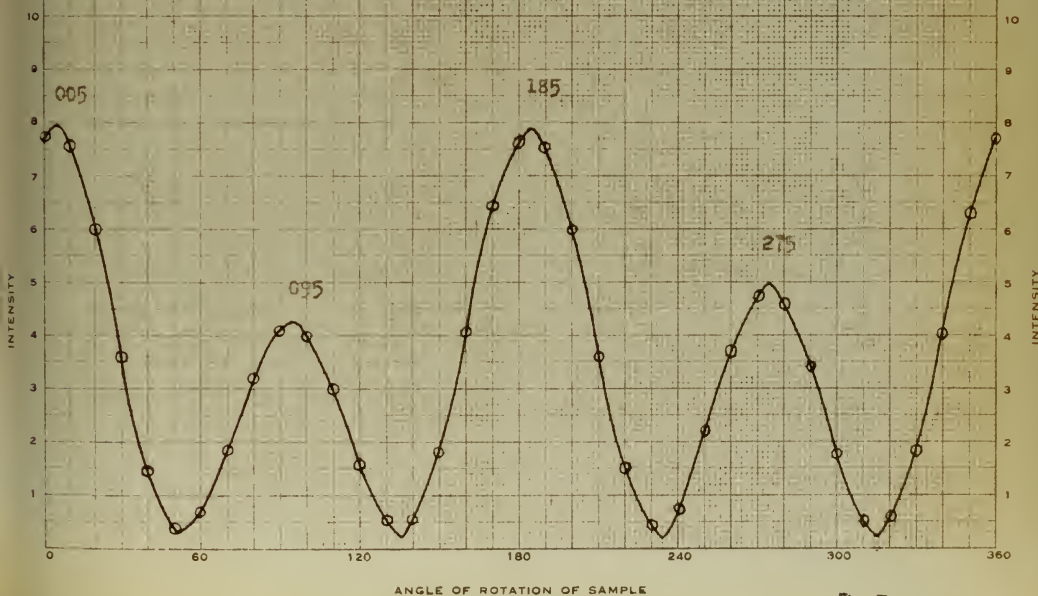
PHASE SHIFT

AV  $\Delta R$  -.58

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 11

PPM 11

DATE 24 MARCH 1966

SURFICIAL NO 2

SURFACE FILM CONDITION

DATE OF FILM 11 MARCH 1966

@ 025		@ 115		@ 200		@ 295	
M	R	M	R	M	R	M	R
44.3	45.1	34.0	44.6	49.6	44.9	34.4	46.2
49.5	45.3	34.5	46.5	46.3	45.4	34.7	46.4
AV 49.10	45.20	34.25	45.55	48.45	45.15	34.55	46.30

M	49.10	R	45.20
M	34.25	R	45.55
M	14.25	ΔR	-1.35

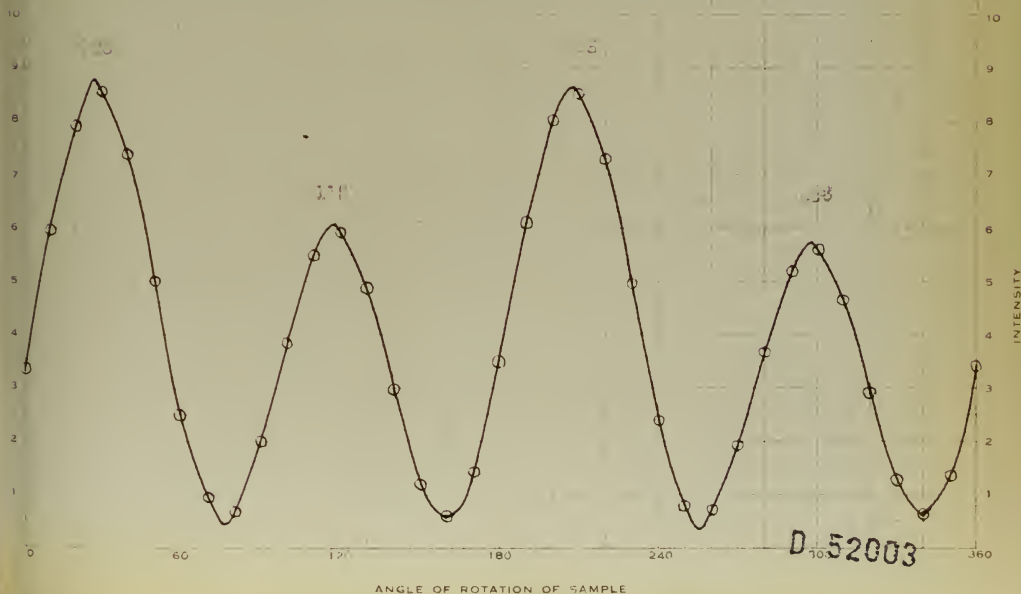
M	49.45	R	45.15
M	34.55	R	45.30
M	14.20	ΔR	-1.15

AV ΔM 14.98 PHASE SHIFT

AV ΔR -1.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CORROSION FILM CONDITION

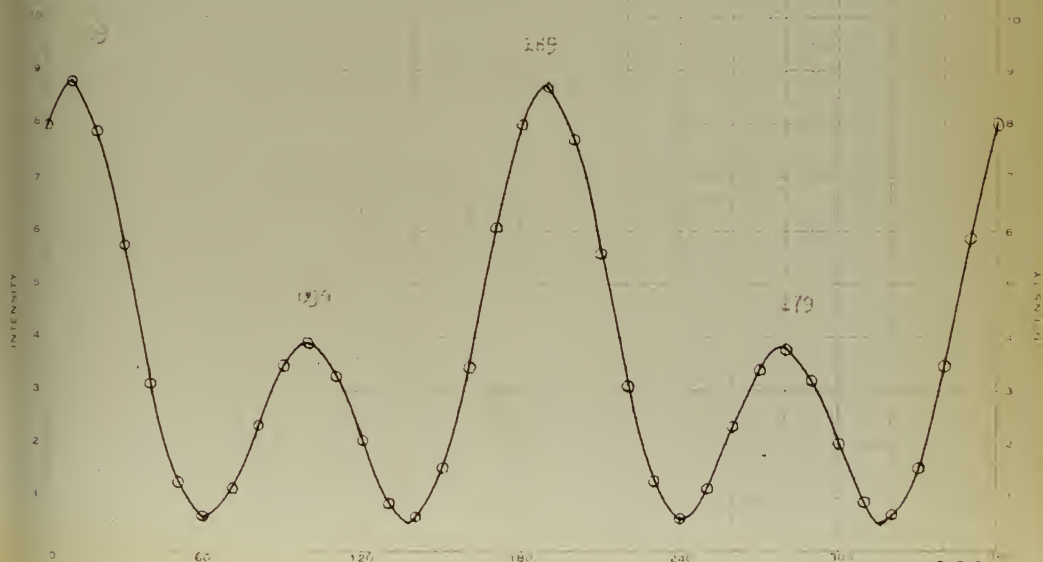
@ 000		@ 099		@ 189		@ 279	
M	R	M	R	M	R	M	R
51.3	45.0	34.4	46.4	50.7	44.0	35.1	44.2
51.3	45.0	34.4	46.4	50.7	44.0	35.1	44.2
AV 51.30	45.00	34.40	46.10	50.70	44.80	35.05	44.70

M	51.30	R	45.00
M	34.80	R	46.10
ΔM	16.50	ΔR	-1.10

M	50.75	R	44.35
M	35.05	R	46.30
ΔM	15.70	ΔR	-1.45

AV ΔM 16.10 PHASE SHIFT  
 AV ΔR -1.28 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE





SAMPLE No 13

150 PPM  $N_2$ 

DATE 25 MARCH 1952 AM

CRYSTAL No 1

SURFACE FILM CONDITION

7th AUTOCLAVE OF 15 MIN. at 233°C

@ 043		@ 133		@ 223		@ 313	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.9	44.9	34.7	46.1	52.0	44.2	34.8	45.9
51.8	44.8	34.8	46.1	51.9	44.6	35.0	46.0
51.85	44.85	34.75	46.10	51.95	44.40	34.90	45.95

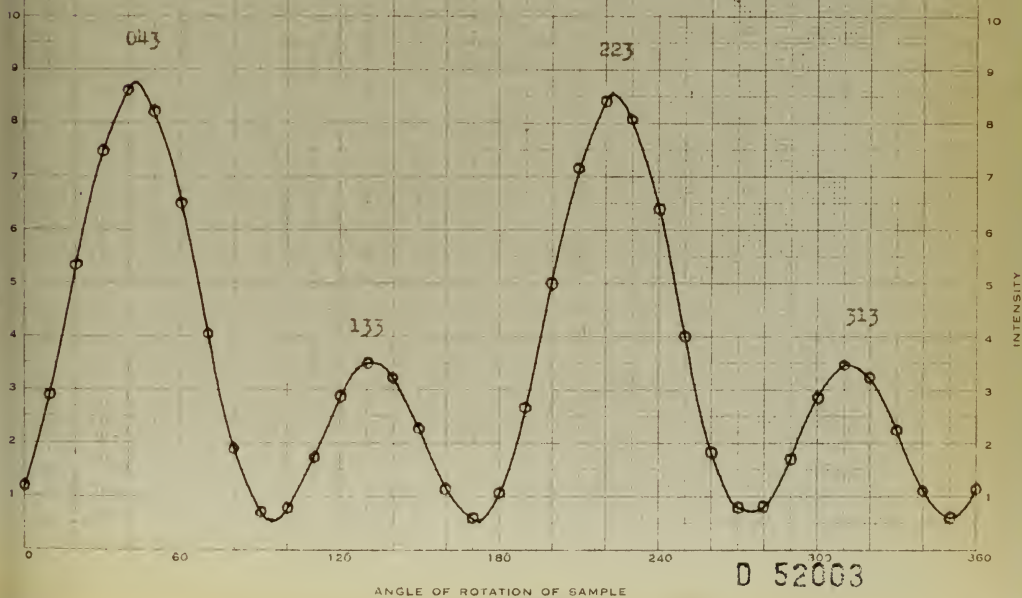
M <sub>1</sub>	51.85	R <sub>1</sub>	44.85
M <sub>2</sub>	34.75	R <sub>2</sub>	46.10
$\Delta M_{12}$	17.10	$\Delta R_{12}$	-1.25

M <sub>3</sub>	51.95	R <sub>3</sub>	44.40
M <sub>4</sub>	34.90	R <sub>4</sub>	45.95
$\Delta M_{34}$	17.05	$\Delta R_{34}$	-1.55

AV  $\Delta M$  17.08 PHASE SHIFTAV  $\Delta R$  -1.40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE No 1E

150 PPM  $N_2$ 

DATE 27 MARCH 1952 PM

CRYSTAL No 1

SURFACE FILM CONDITION

8th AUTOCLAVE CP 15 MIN. @ 233°C

@ 081		@ 171		@ 261		@ 351	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.9	44.5	33.4	46.3	51.3	44.6	33.7	46.0
51.7	44.9	33.6	46.2	51.4	44.6	33.3	46.4
Av 51.80	44.70	33.50	46.25	51.35	44.60	33.50	46.20

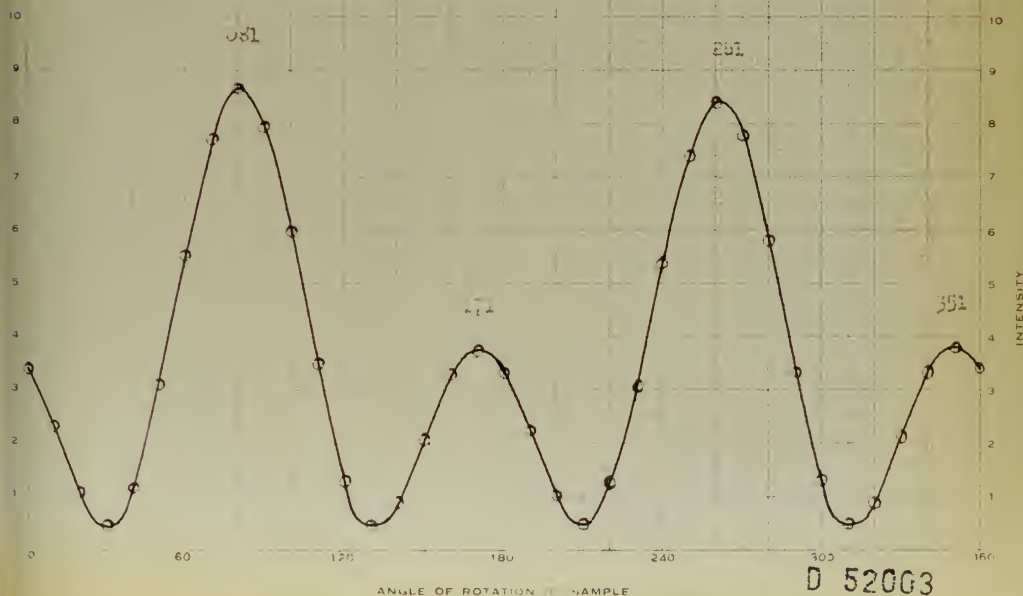
M <sub>1</sub> 51.80	R <sub>1</sub> 44.70
M <sub>2</sub> 33.50	R <sub>2</sub> 46.25
$\Delta M_1$ 18.30	$\Delta R_1$ -1.55

M <sub>3</sub> 51.35	R <sub>3</sub> 44.60
M <sub>4</sub> 33.50	R <sub>4</sub> 46.20
$\Delta M_{34}$ 17.80	$\Delta R_{34}$ -1.60

Av  $\Delta M$  18.05 PHASE SHIFTAv  $\Delta R$  -1.58 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 520G3



CRYSTAL No 1 SURFACE FILM CONDITION 9th AUTOCLAVE OF 15 MIN. @ 233°C

@ 330		@ 060		@ 150		@ 240	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.9	44.8	35.7	45.9	52.2	45.1	35.2	46.0
52.6	44.6	35.5	46.2	51.9	45.0	34.8	46.3
52.75	44.70	35.60	46.05	52.05	45.05	35.00	46.15

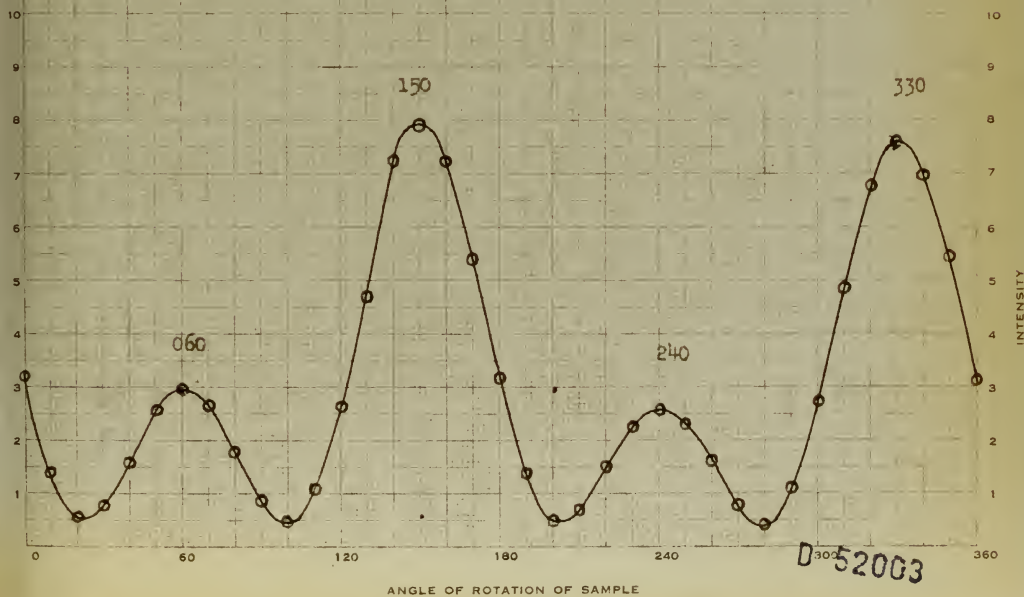
M<sub>1</sub> 52.75 R<sub>1</sub> 44.70  
M<sub>2</sub> 35.60 R<sub>2</sub> 46.05  
ΔM<sub>12</sub> 17.15 ΔR<sub>12</sub> -1.35

M<sub>3</sub> 52.05 R<sub>3</sub> 45.05  
M<sub>4</sub> 35.00 R<sub>4</sub> 46.15  
ΔM<sub>34</sub> 17.05 ΔR<sub>34</sub> -1.10

AV ΔM 17.10 PHASE SHIFT  
AV ΔR -1.23 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 13

150 PPM N<sub>2</sub>

DATE 30 MARCH 1952 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

10th AUTOCLAVE OF 15 MIN. @ 233°C

AV

@ 031		@ 121		@ 211		@ 301	
M	R	M	R	M	R	M	R
52.3	45.0	34.6	46.6	51.9	44.6	33.7	46.2
52.5	44.8	34.2	46.5	52.0	44.6	33.7	46.5
52.40	44.90	34.40	46.60	51.95	44.60	33.70	46.35

M <sub>1</sub> 52.40	R <sub>1</sub> 44.90
M <sub>2</sub> 34.40	R <sub>1</sub> 46.60
$\Delta M_2$ 18.00	$\Delta R_2$ -1.70

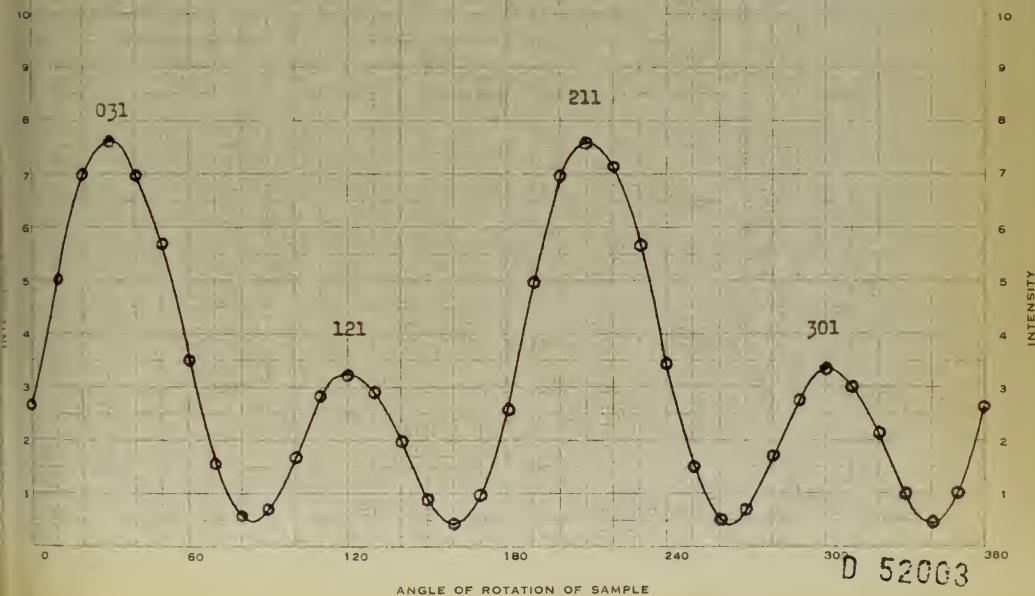
  

M <sub>1</sub> 51.95	R <sub>1</sub> 44.60
M <sub>2</sub> 33.70	R <sub>1</sub> 46.35
$\Delta M_{31}$ 18.25	$\Delta R_{31}$ -1.75

AV  $\Delta M$  18.13 PHASE SHIFTAV  $\Delta R$  -1.73 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE NO 1E

150 PPM N<sub>2</sub>

DATE 1 APRIL 1952 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

11th AUTOCLAVE OF 15 MIN. @ 233°C

@ 343		@ 073		@ 163		@ 253	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.2	45.0	33.7	46.1	50.4	45.1	33.5	46.0
51.2	45.0	33.4	46.0	51.0	44.8	33.0	45.9
51.20	45.00	33.55	46.05	50.70	44.95	33.25	45.95

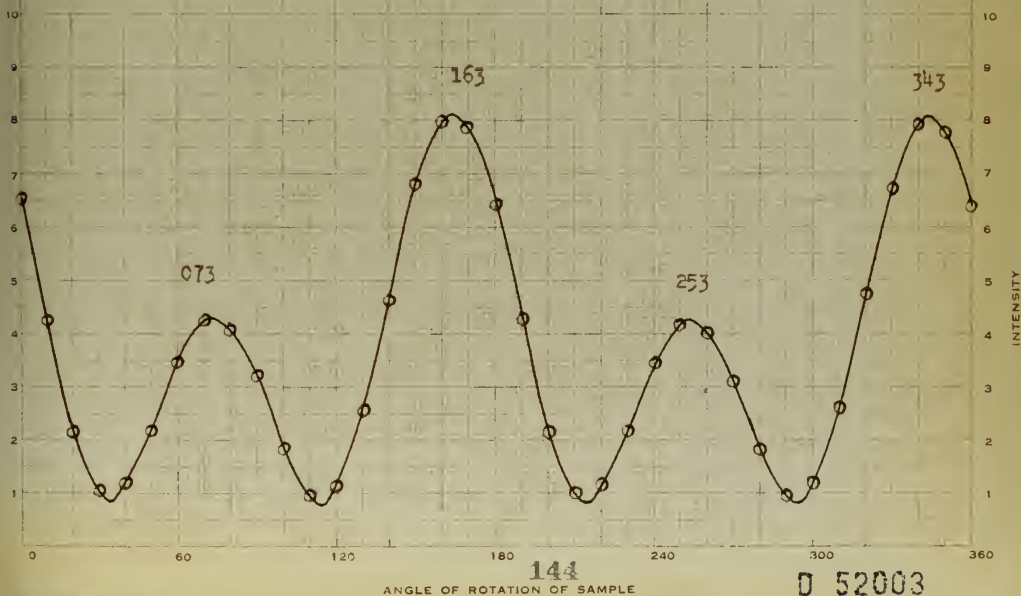
M <sub>1</sub>	51.20	R <sub>1</sub>	45.00
M <sub>2</sub>	33.55	R <sub>2</sub>	46.05
$\Delta M_{12}$	17.65	$\Delta R_{12}$	-1.05

M <sub>3</sub>	50.70	R <sub>3</sub>	44.95
M <sub>4</sub>	33.25	R <sub>4</sub>	45.95
$\Delta M_{34}$	17.45	$\Delta R_{34}$	-1.00

AV  $\Delta M$  17.55 PHASE SHIFTAV  $\Delta R$  -1.05 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



CRYSTAL No 1 SURFACE FILM CONDITION 12th AUTOCLAVE OF 15 MIN. @ 233°C

@ 328		@ 258		@ 148		@ 238	
M	R	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.7	44.7	33.6	46.3	50.0	44.9	33.0	46.0
50.7	44.8	33.5	46.2	49.6	44.7	33.0	46.0
50.70	44.75	33.55	46.25	49.80	44.80	33.00	46.00

M <sub>1</sub>	50.70	R <sub>1</sub>	44.75
M <sub>2</sub>	33.55	R <sub>2</sub>	46.25
$\Delta M_2$	17.15	$\Delta R_2$	-1.50

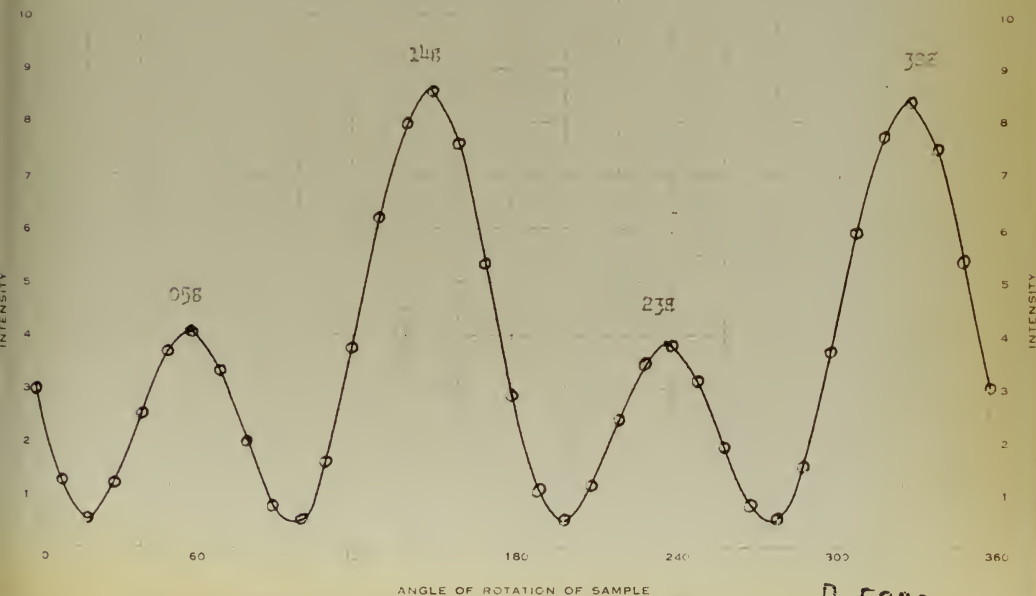
M <sub>3</sub>	49.80	R <sub>3</sub>	44.80
M <sub>4</sub>	33.00	R <sub>4</sub>	46.00
$\Delta M_{34}$	16.80	$\Delta R_{34}$	-1.20

AV  $\Delta M$  16.98 PHASE SHIFT

AV  $\Delta R$  -1.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE No 17

150 PPM  $H_2$ 

DATE 3 APR 1967 BW

CRYSTAL No 1

SURFACE FILM CONDITION

150A ROTATION OF 15 MIN. at 233°C

@ 056		@ 146		@ 236		@ 326	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.2	45.2	33.7	46.2	50.4	44.8	33.4	46.3
51.2	44.8	53.7	46.5	50.8	44.9	33.4	46.4
AV 51.20	45.00	33.70	46.35	50.60	44.85	33.50	46.35

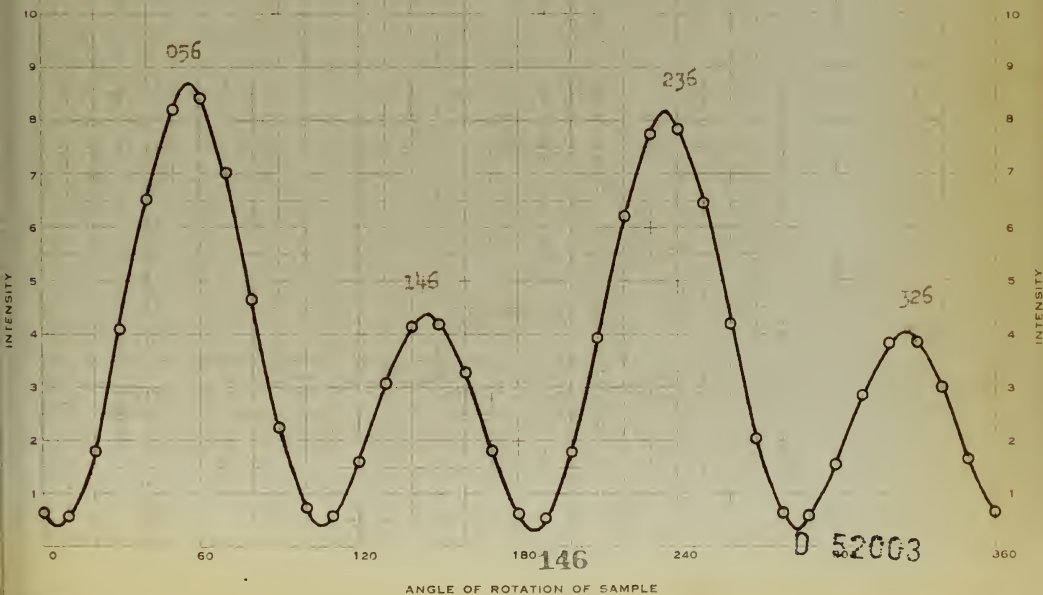
M <sub>1</sub>	51.20	R <sub>1</sub>	45.00
M <sub>2</sub>	33.70	R <sub>2</sub>	46.35
$\Delta M_{12}$	17.50	$\Delta R_{12}$	-1.35

M <sub>3</sub>	50.60	R <sub>3</sub>	44.85
M <sub>4</sub>	33.50	R <sub>4</sub>	46.35
$\Delta M_{34}$	17.20	$\Delta R_{34}$	-1.50

AV  $\Delta M$  17.55 PHASE SHIFTAV  $\Delta R$  -1.43 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE No. 13

150 PPM  $N_2$ 

DATE 5 APRIL 1952 PM

CRYSTAL No. 1

SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 063		@ 153		@ 243		@ 333	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
52.2	44.5	34.6	46.3	51.3	44.7	34.5	46.2
52.4	44.7	34.6	46.4	51.8	44.5	34.5	46.4
AV 52.30	44.60	34.60	46.35	51.55	44.60	34.50	46.30

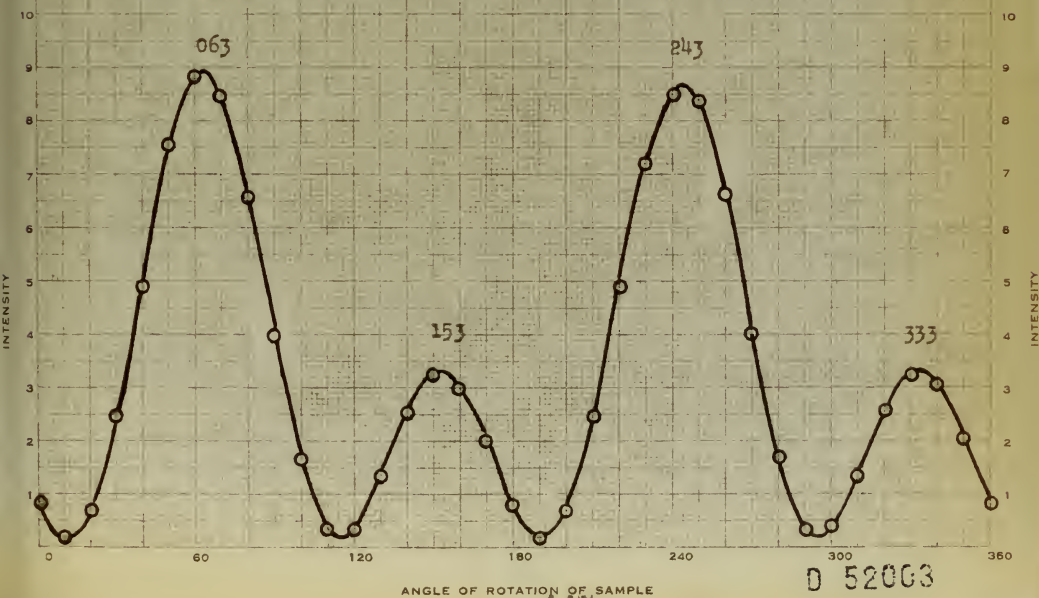
M <sub>1</sub>	52.30	R <sub>1</sub>	44.60
M <sub>2</sub>	34.60	R <sub>2</sub>	46.35
$\Delta M_{12}$	17.70	$\Delta R_{12}$	-1.75

M <sub>3</sub>	51.55	R <sub>3</sub>	44.60
M <sub>4</sub>	34.50	R <sub>4</sub>	46.30
$\Delta M_{34}$	17.05	$\Delta R_{34}$	-1.70

AV  $\Delta M$  17.38 PHASE SHIFTAV  $\Delta R$  -1.73 PHASE ROTATION

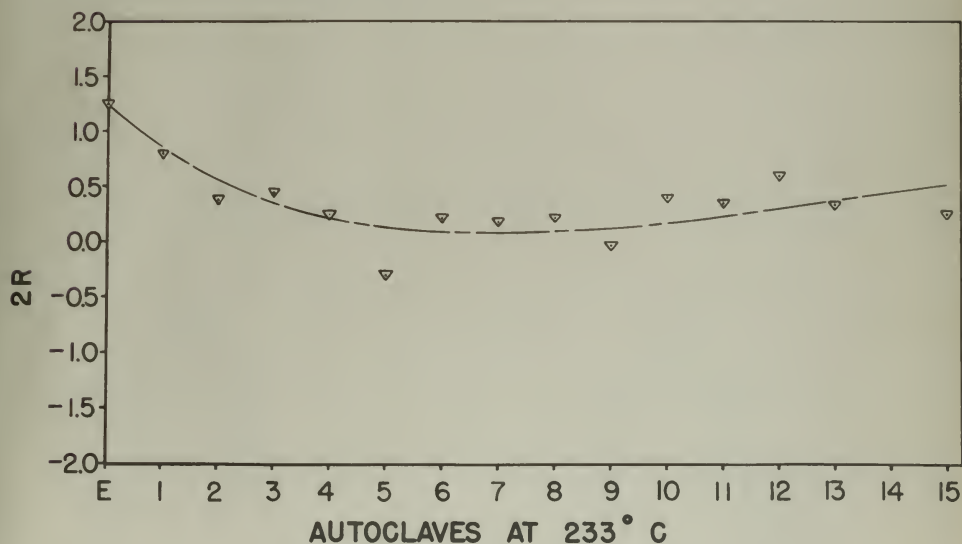
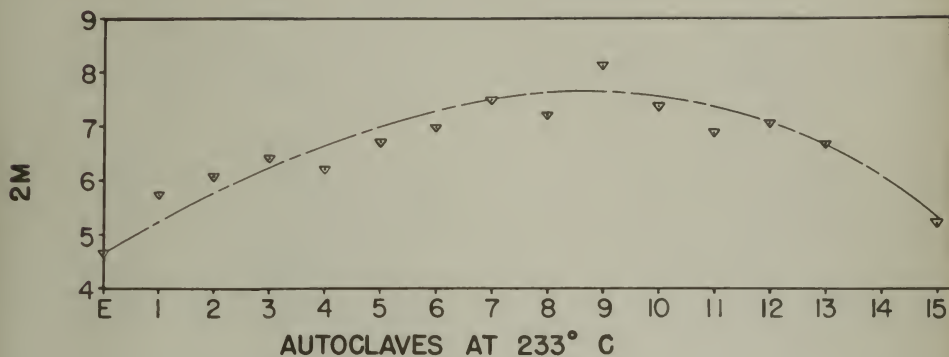
PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





PHASE SHIFT (2m) AND ROTATION OF PLANE OF  
POLARIZATION (2r) VS. CORROSION  
TIME FOR SAMPLE NO. 1F-1



CRYSTAL No 1 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 256°		@ 166°		@ 67°		@ 346°	
	M1	R1	M2	R2	M3	R3	M4	R4
	45.1	46.1	40.3	45.0	44.9	46.1	40.5	44.7
	45.3	46.1	40.3	45.0	44.9	46.2	40.5	44.8
AV.	45.20	46.10	40.30	45.00	44.90	46.15	40.5	44.75

M <sub>1</sub>	45.20	R <sub>1</sub>	46.10
M <sub>2</sub>	40.30	R <sub>2</sub>	45.00
$\Delta M_{12}$	4.90	$\Delta R_{12}$	1.10

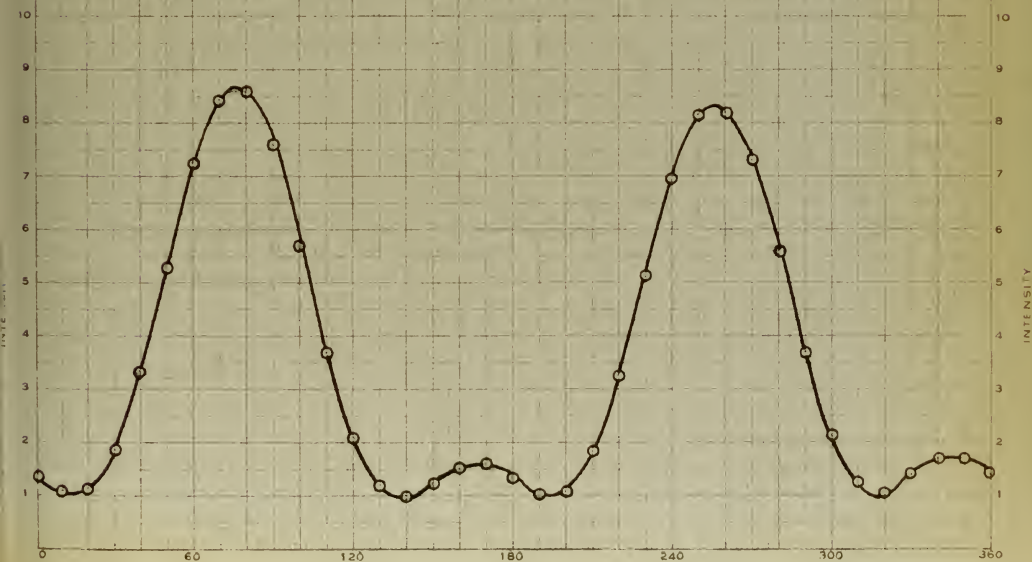
M <sub>3</sub>	44.9	R <sub>3</sub>	46.15
M <sub>4</sub>	40.5	R <sub>4</sub>	44.75
$\Delta M_{34}$	4.40	$\Delta R_{34}$	1.40

AV  $\Delta M$  4.65 PHASE SHIFT

AV  $\Delta R$  1.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 1F

PPM 2

DATE 12 MARCH 1952 AV

CRYSTAL NO 1

SURFACE FILM CONDITION

1st AUTOCLAVE 15 MIN. at 231°C

@ 006		@ 275		@ 186		@ 096	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.2	45.9	39.2	45.1	45.1	45.9	39.5	45.1
45.0	45.9	39.4	45.1	45.0	45.9	39.3	45.1
AV. 45.10	45.90	39.30	45.10	45.05	45.90	39.40	45.10

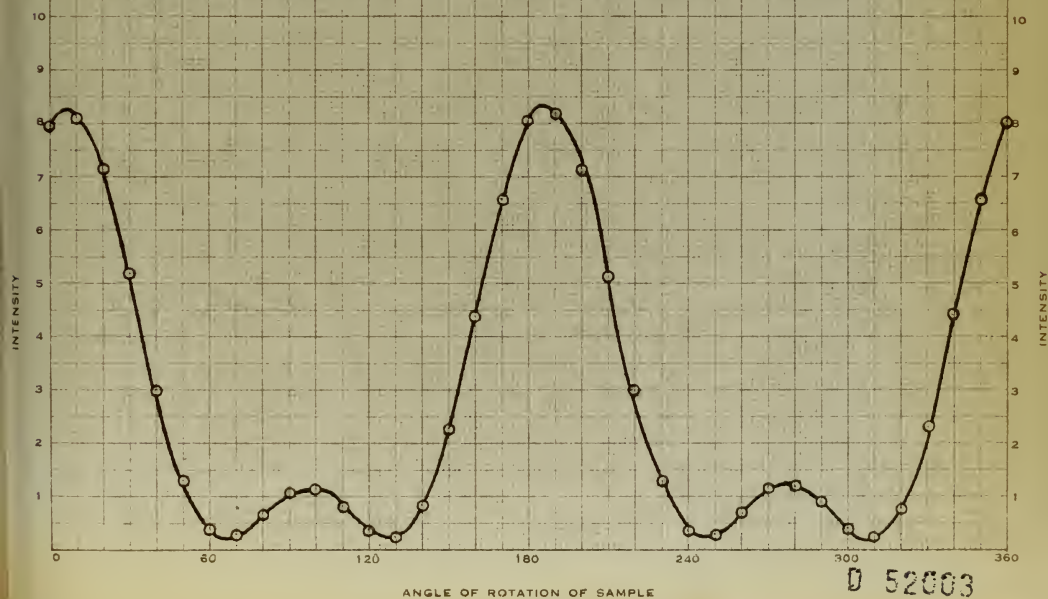
M <sub>1</sub>	45.10	R <sub>1</sub>	45.90
M <sub>2</sub>	39.30	R <sub>2</sub>	45.10
$\Delta M_{12}$	5.80	$\Delta R_{12}$	0.80

M <sub>3</sub>	45.05	R <sub>3</sub>	45.90
M <sub>4</sub>	39.40	R <sub>4</sub>	45.10
$\Delta M_{34}$	5.65	$\Delta R_{34}$	0.30

AV  $\Delta M$  5.72 PHASE SHIFTAV  $\Delta R$  0.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL NO. 1 SURFACE FILM CONDITION 2nd AUTOCLAVE 15 MIN. at 233°C

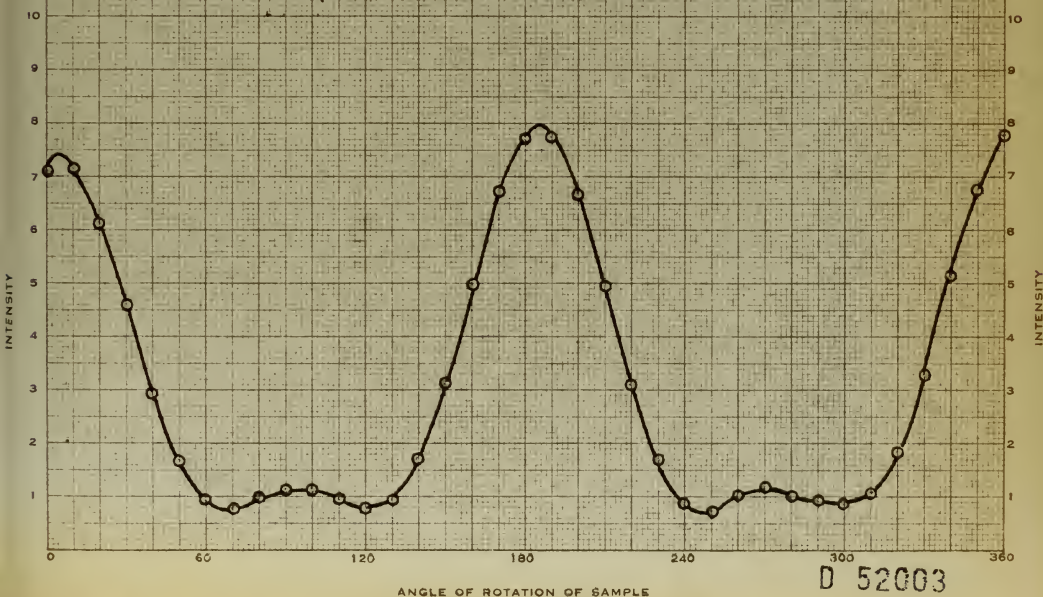
@ 005		@ 275		@ 185		@ 095	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.4	45.5	40.2	45.2	46.2	45.6	40.2	45.2
46.4	45.4	40.2	45.2	46.1	45.7	40.2	45.2
AV. 46.40	45.50	40.20	45.20	46.15	45.65	40.20	45.20

M <sub>1</sub>	46.40	R <sub>1</sub>	45.30
M <sub>2</sub>	40.20	R <sub>2</sub>	45.30
$\Delta M_{12}$	6.20	$\Delta R_{12}$	0.30

M <sub>3</sub>	46.15	R <sub>3</sub>	45.65
M <sub>4</sub>	40.20	R <sub>4</sub>	45.20
$\Delta M_{34}$	5.95	$\Delta R_{34}$	0.45

AV  $\Delta M$  6.08 PHASE SHIFT  
 AV  $\Delta R$  0.38 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



CRYSTAL No 1

SURFACE FILM CONDITION

3rd AUTOCLAVE 15 MIN. at 233°C

@ 333		@ 243		@ 153		@ 63	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.2	45.7	39.9	45.3	46.30	45.80	39.7	45.2
46.3	45.7	40.1	45.3	46.20	45.70	39.7	45.3
AV 46.25	45.70	40.00	45.30	46.25	45.75	39.70	45.25

M <sub>1</sub>	46.25	R <sub>1</sub>	45.70
M <sub>2</sub>	40.50	R <sub>2</sub>	45.30
$\Delta M_{12}$	6.25	$\Delta R_{12}$	0.40

M <sub>3</sub>	46.25	R <sub>3</sub>	45.75
M <sub>4</sub>	39.70	R <sub>4</sub>	45.25
$\Delta M_{34}$	6.55	$\Delta R_{34}$	0.50

AV  $\Delta M$  6.40

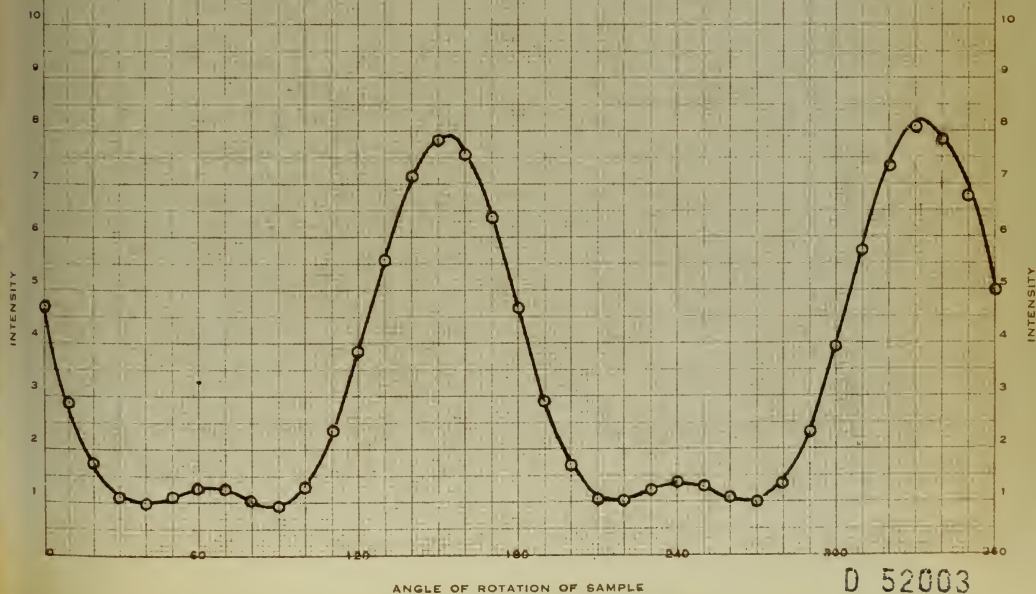
PHASE SHIFT

AV  $\Delta R$  0.45

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL NO 1

SURFACE FILM CONDITION

4th AUTOCLAVE 15 MIN. @ 233°C

@ 282		@ 192		@ 102		@ 012	
M1	R1	M2	R2	M3	R3	M4	R4
45.6	45.8	39.5	45.7	45.6	45.9	39.2	45.7
45.6	46.0	39.6	45.7	45.4	46.1	39.0	45.7
AV. 45.60	45.90	39.60	45.70	45.50	46.00	39.10	45.70

M1	45.60	R1	45.90
M2	39.60	R2	45.70
$\Delta M_{12}$	6.00	$\Delta R_{12}$	0.20

M3	45.50	R3	46.00
M4	39.10	R4	45.70
$\Delta M_{34}$	6.40	$\Delta R_{34}$	0.30

AV  $\Delta M$  6.20

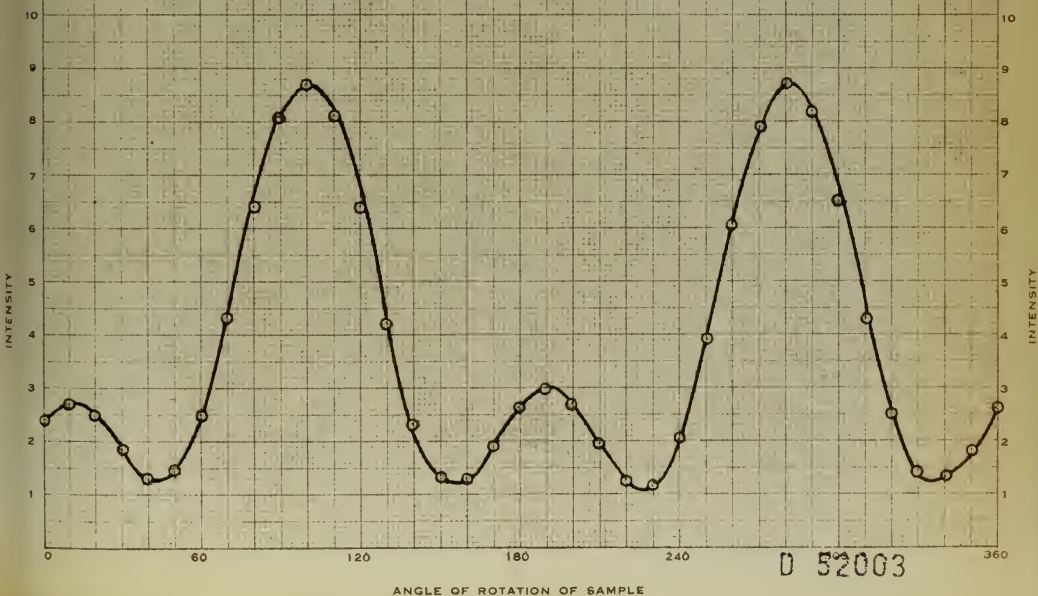
PHASE SHIFT

AV  $\Delta R$  0.25

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 1F

PPM

DATE 24 MARCH 1962 PM

CRYSTAL NO 1

SURFACE FILM CONDITION

5th AUTOCLAVE 15 MIN @ 233°C

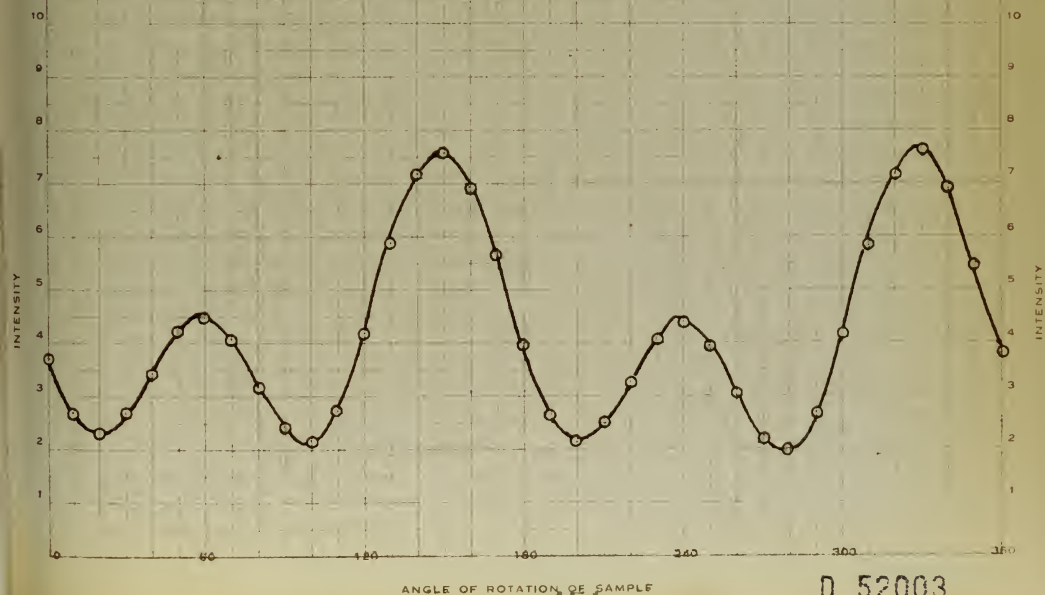
	@ 327		@ 337		@ 347		@ 357	
	M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
	45.0	46.0	38.2	45.8	44.7	46.0	38.0	45.7
	45.0	46.0	38.4	45.7	44.7	46.0	38.0	45.6
AV.	45.00	46.00	38.30	45.75	44.70	46.00	38.00	45.65

M <sub>1</sub>	45.00	R <sub>1</sub>	46.00
M <sub>2</sub>	38.30	R <sub>2</sub>	45.75
$\Delta M_{12}$	6.70	$\Delta R_{12}$	-0.25

M <sub>3</sub>	44.70	R <sub>3</sub>	46.00
M <sub>4</sub>	38.00	R <sub>4</sub>	45.65
$\Delta M_{34}$	6.70	$\Delta R_{34}$	-0.35

AV  $\Delta M$  6.70 PHASE SHIFT  
 AV  $\Delta R$  -0.30 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE



CRYSTAL NO 2

SURFACE FILM CONDITION

6th AUTOCLAVE 15 MIN. @ 233°C

@ 026		@ 296		@ 206		@ 116	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.8	46.0	41.0	45.6	47.7	45.9	40.4	45.7
47.7	46.0	41.0	45.7	47.5	45.8	40.4	45.8
Av 47.75	46.00	41.00	45.65	47.60	45.85	40.40	45.75

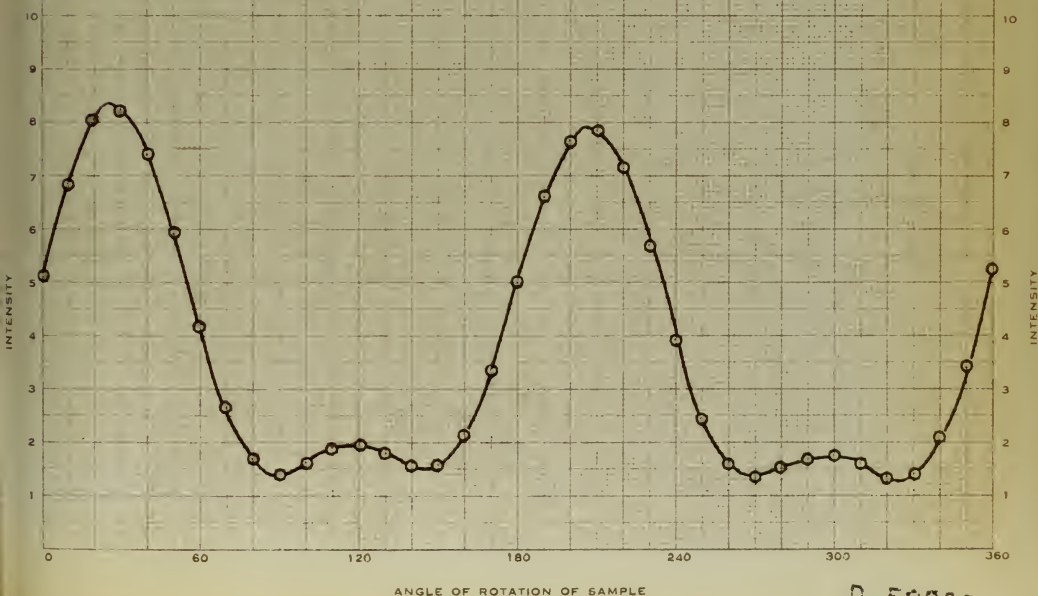
M <sub>1</sub>	47.75	R <sub>1</sub>	46.00
M <sub>2</sub>	41.00	R <sub>2</sub>	45.65
$\Delta M_{12}$	6.75	$\Delta R_{12}$	0.35

M <sub>3</sub>	47.60	R <sub>3</sub>	45.85
M <sub>4</sub>	40.40	R <sub>4</sub>	45.75
$\Delta M_{34}$	7.20	$\Delta R_{34}$	0.10

AV  $\Delta M$  6.97 PHASE SHIFTAV  $\Delta R$  0.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE





CRYSTAL No. 1 SURFACE FILM CONDITION 7th AUTOCLAVE 15 MIN. @ 233°C

@ 115		@ 225		@ 135		@ 045	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.3	45.5	39.8	45.4	47.1	45.6	39.7	45.4
47.4	45.6	39.7	45.4	47.1	45.6	39.8	45.4
AV 47.35	45.55	39.75	45.40	47.10	45.60	39.75	45.40

M<sub>1</sub> 47.35 R<sub>1</sub> 45.55  
M<sub>2</sub> 39.75 R<sub>2</sub> 45.40  
ΔM<sub>12</sub> 7.60 ΔR<sub>12</sub> 0.15

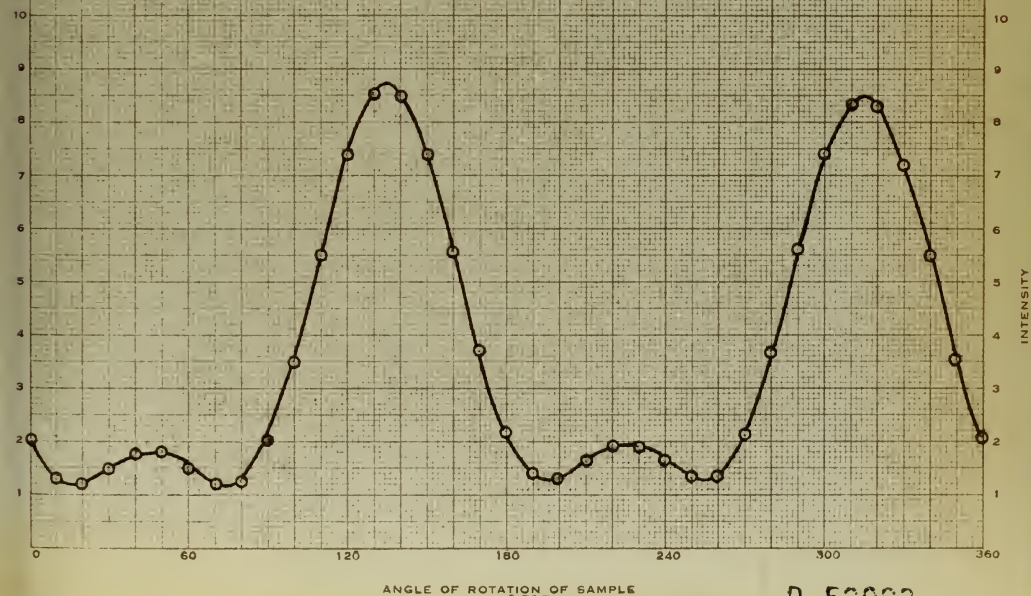
M<sub>3</sub> 47.10 R<sub>3</sub> 45.60  
M<sub>4</sub> 39.75 R<sub>4</sub> 45.40  
ΔM<sub>34</sub> 7.35 ΔR<sub>34</sub> 0.20

AV ΔM 7.48 PHASE SHIFT

AV ΔR 0.18 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CRYSTAL No. 1

SURFACE FILM CONDITION

in AUTOCLAVE 15 MIN. at 23.0°C

@ 039.....		@ 399.....		@ 219.....		@ 129.....	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.9	45.5	39.7	45.3	46.8	45.6	39.7	45.3
47.0	45.5	39.6	45.4	46.7	45.6	39.6	45.3
AV 46.95	45.50	39.65	45.35	46.75	45.60	39.65	45.30

M <sub>1</sub>	46.95	R <sub>1</sub>	45.50
M <sub>2</sub>	39.65	R <sub>2</sub>	45.35
$\Delta M_{12}$	7.30	$\Delta R_{12}$	0.15

M <sub>3</sub>	46.75	R <sub>3</sub>	45.60
M <sub>4</sub>	39.65	R <sub>4</sub>	45.30
$\Delta M_{34}$	7.10	$\Delta R_{34}$	0.30

AV  $\Delta M$  7.20

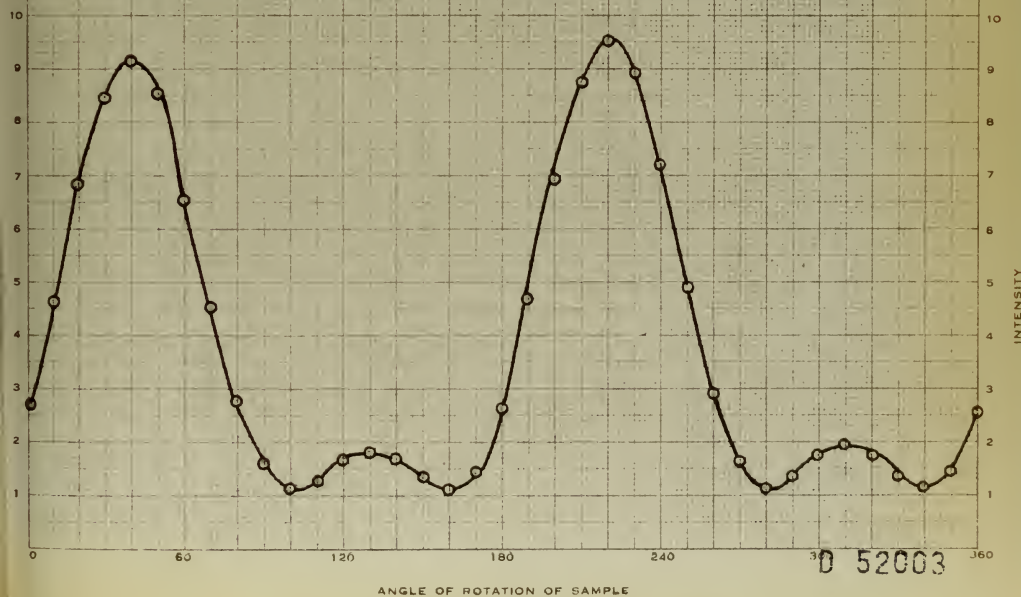
PHASE SHIFT

AV  $\Delta R$  0.22

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 1F

24 PPM 2

DATE 28 MARCH 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION 9th AUTOCLAVE 15 MIN @ 233°C

@ 028		@ 298		@ 208		@ 118	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
48.1	45.6	39.6	45.1	47.7	45.6	39.8	45.4
47.8	45.4	39.1	45.7	47.7	45.5	39.7	45.4
AV 47.95	45.50	39.65	45.70	47.70	45.55	39.75	45.40

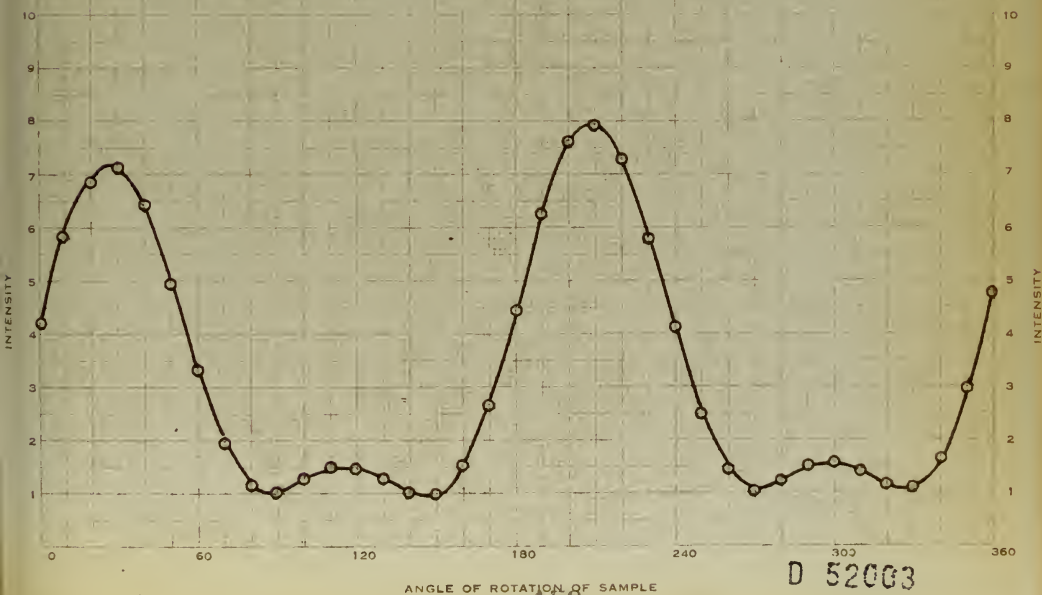
M <sub>1</sub>	47.95	R <sub>1</sub>	45.50
M <sub>2</sub>	39.65	R <sub>2</sub>	45.70
$\Delta M_{12}$	8.30	$\Delta R_2$	-0.20

M <sub>3</sub>	47.70	R <sub>3</sub>	45.55
M <sub>4</sub>	39.75	R <sub>4</sub>	45.40
$\Delta M_{34}$	7.95	$\Delta R_{34}$	0.15

AV  $\Delta M$  8.12 PHASE SHIFTAV  $\Delta R$  -0.03 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE NO. 17

240 PPM N<sub>2</sub>

DATE 31 MARCH 1952 AM

CRYSTAL NO. 1

SURFACE FILM CONDITION

10th AUTOCLAVE 15 MIN. @ 233°C

@ 225		@ 135		@ 045		@ 315	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.7	46.0	40.5	45.5	47.5	45.9	40.0	45.6
47.7	46.1	40.5	45.7	47.6	45.8	40.0	45.4
47.70	46.05	40.50	45.60	47.55	45.85	40.00	45.50

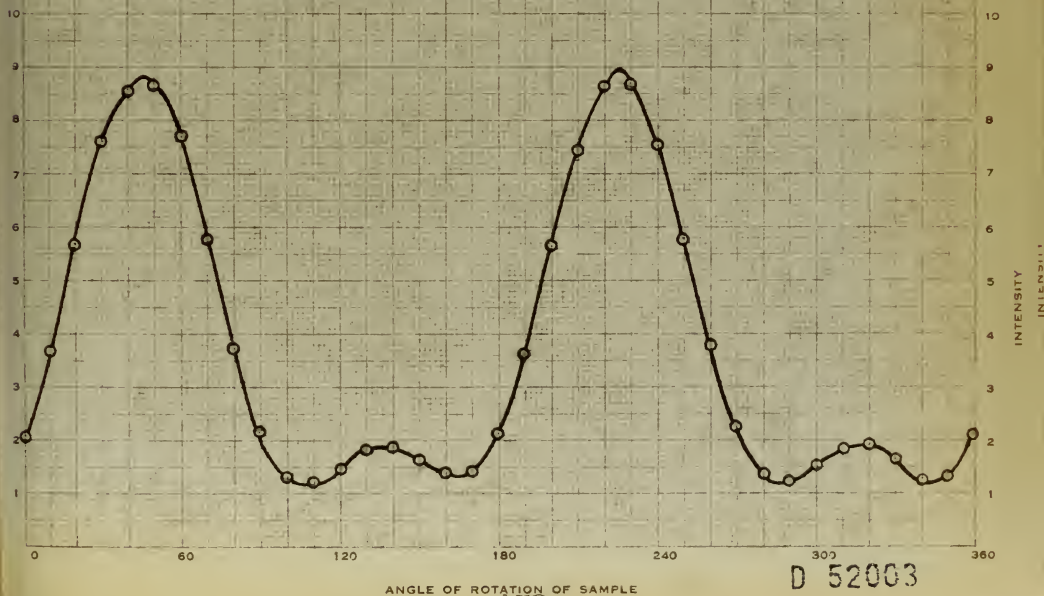
M <sub>1</sub>	47.70	R <sub>1</sub>	46.05
M <sub>2</sub>	40.50	R <sub>2</sub>	45.60
$\Delta M_{12}$	7.20	$\Delta R_{12}$	0.45

M <sub>3</sub>	47.55	R <sub>3</sub>	45.85
M <sub>4</sub>	40.00	R <sub>4</sub>	45.50
$\Delta M_{34}$	7.55	$\Delta R_{34}$	0.35

AV  $\Delta M$  7.37 PHASE SHIFTAV  $\Delta R$  0.40 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE No 1F

240 PPM N<sub>2</sub>

DATE 1 APRIL 1952 AM

CRYSTAL No 1

SURFACE FILM CONDITION

11th AUTOCLAVE 15 MIN. @ 233°C

@ 326		@ 236		@ 146		@ 056	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.5	45.6	38.7	45.4	45.5	45.8	38.5	45.4
45.3	45.7	38.6	45.3	45.5	45.7	38.5	45.3
AV 45.40	45.65	38.65	45.35	45.50	45.75	38.50	45.35

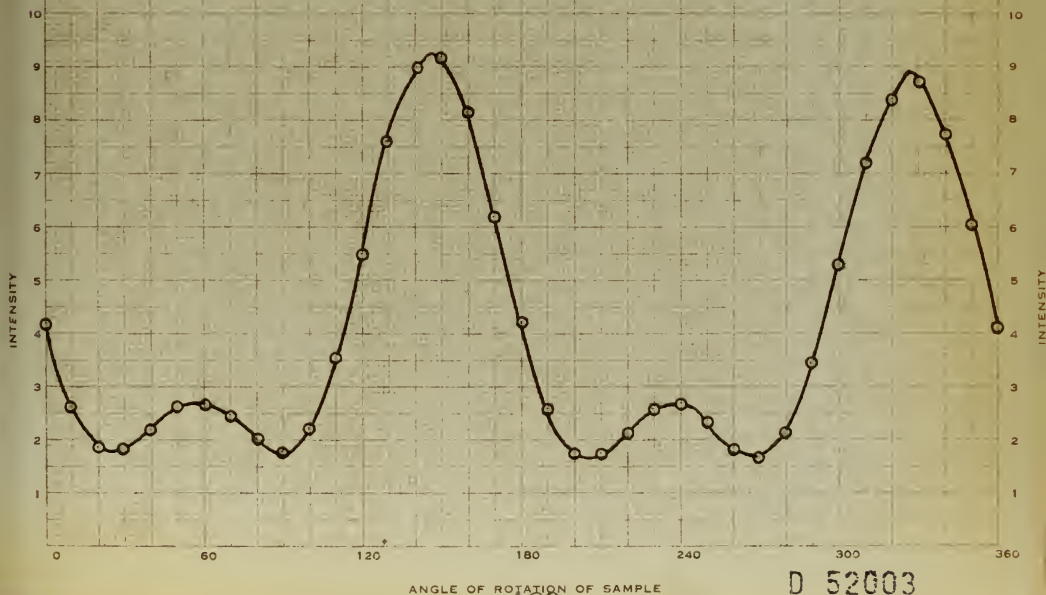
M <sub>1</sub>	45.40	R <sub>1</sub>	45.65
M <sub>2</sub>	38.65	R <sub>2</sub>	45.35
$\Delta M_{12}$	6.75	$\Delta R_{12}$	0.30

M <sub>3</sub>	45.50	R <sub>3</sub>	45.75
M <sub>4</sub>	38.50	R <sub>4</sub>	45.35
$\Delta M_{34}$	7.00	$\Delta R_{34}$	0.40

AV  $\Delta M$  6.87 PHASE SHIFTAV  $\Delta R$  0.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE NO 17

240 PPM  $\lambda_2$ 

DATE 2 APRIL 1952 AM

CRYSTAL NO 1

SURFACE FILM CONDITION

12th AUTOCLAVE 15 MIN @ 233°C

@ 252		@ 132		@ 642		@ 312	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.7	45.9	38.3	45.1	45.4	45.5	38.7	45.2
45.4	45.7	38.0	45.0	45.4	45.7	32.8	45.1
AV. 45.65	45.80	38.15	45.05	45.40	45.60	38.80	45.15

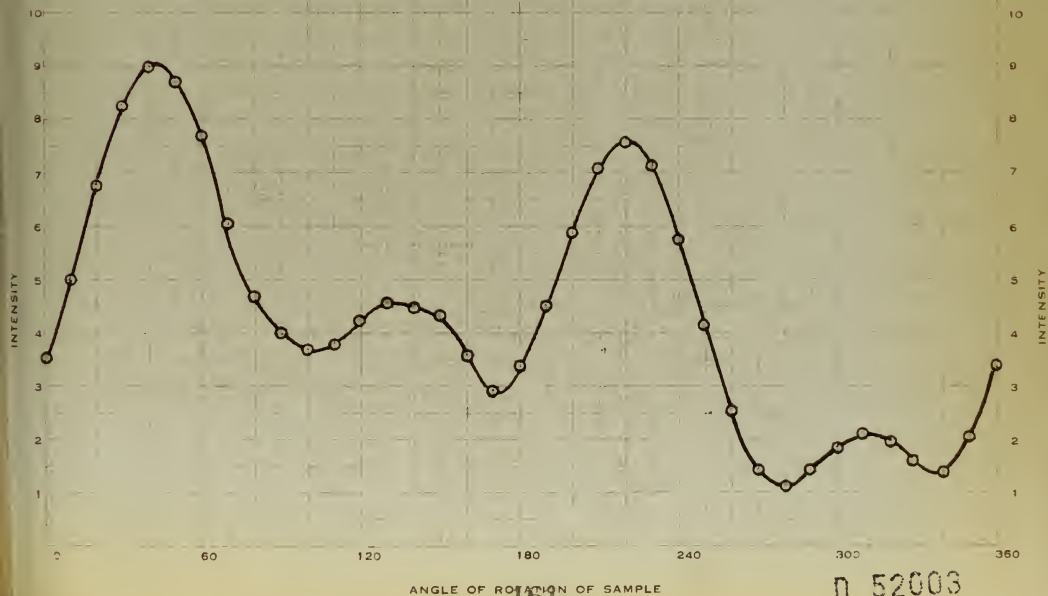
M <sub>1</sub>	45.65	R <sub>1</sub>	45.80
M <sub>2</sub>	38.15	R <sub>2</sub>	45.05
$\Delta M_{12}$	7.50	$\Delta R_{12}$	.75

M <sub>3</sub>	45.40	R <sub>3</sub>	45.60
M <sub>4</sub>	38.80	R <sub>4</sub>	45.15
$\Delta M_{34}$	6.60	$\Delta R_{34}$	.45

AV  $\Delta M$  7.05 PHASE SHIFTAV  $\Delta R$  0.60 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE No. 1F

240 PPM  $N_2$ 

DATE 4 APRIL 1952 AM

CRYSTAL No. 1

SURFACE FILM CONDITION

13th AUTOCLAVE 15 MIN. @ 233°C

@ 210		@ 120		@ 030		@ 300	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.3	46.1	38.9	45.8	45.5	46.0	38.6	45.7
45.5	46.0	38.9	45.6	45.5	46.0	38.7	45.7
Av. 45.40	46.05	38.90	45.70	45.50	46.00	38.65	45.70

M <sub>1</sub> 45.40	R <sub>1</sub> 46.05
M <sub>2</sub> 38.90	R <sub>2</sub> 45.70
$\Delta M_{12}$ 6.50	$\Delta R_{12}$ 0.35

M <sub>3</sub> 45.50	R <sub>3</sub> 46.00
M <sub>4</sub> 38.65	R <sub>4</sub> 45.70
$\Delta M_{34}$ 6.85	$\Delta R_{34}$ 0.30

Av  $\Delta M$  6.67

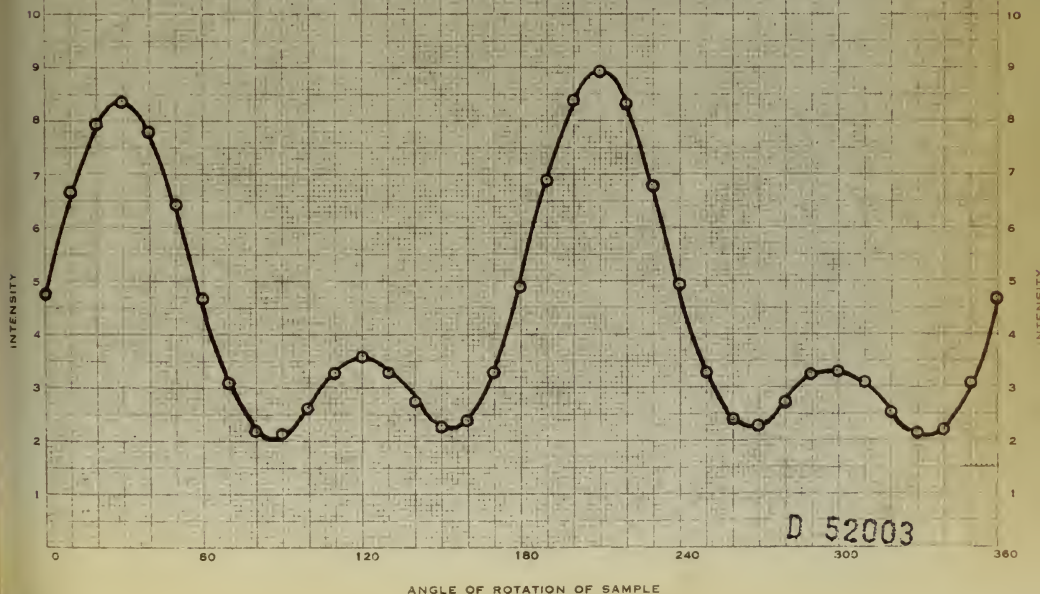
PHASE SHIFT

Av  $\Delta R$  0.33

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL No 1 SURFACE FILM CONDITION 15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 264		@ 171		@ 081		@ 354	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.2	45.8	41.9	45.2	46.2	46.0	41.6	45.8
46.9	46.0	41.8	45.7	46.8	46.0	41.6	45.7
AV 47.05		41.85		46.60		41.60	
45.90		45.75		46.00		45.75	

M <sub>1</sub>	47.05	R <sub>1</sub>	45.90
M <sub>2</sub>	41.85	R <sub>2</sub>	45.75
$\Delta M_{12}$	5.20	$\Delta R_{12}$	0.25

M <sub>3</sub>	46.80	R <sub>3</sub>	46.00
M <sub>4</sub>	41.50	R <sub>4</sub>	45.75
$\Delta M_{34}$	5.20	$\Delta R_{34}$	0.25

AV  $\Delta M$  5.20 PHASE SHIFT  
 AV  $\Delta R$  0.25 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

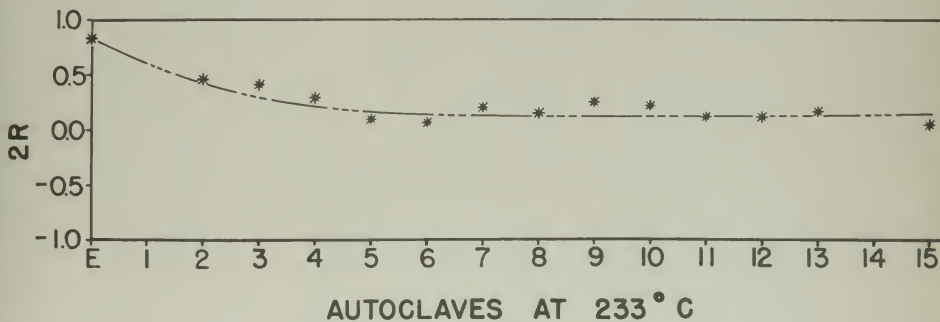
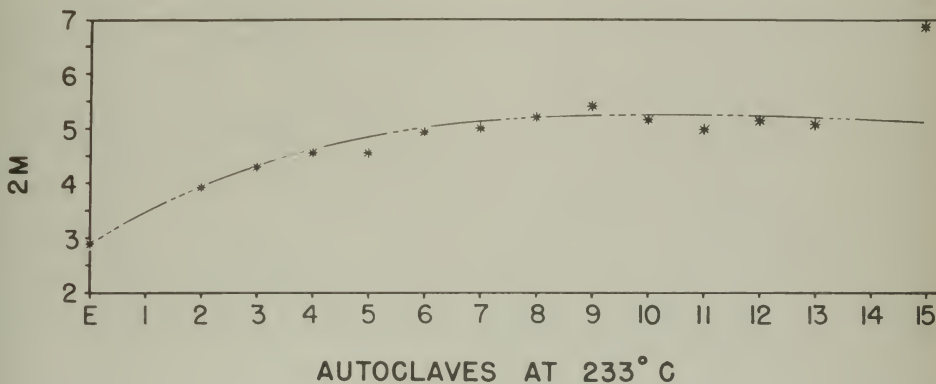
GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003







PHASE SHIFT ( $2m$ ) AND ROTATION OF PLANE  
OF POLARIZATION ( $2r$ ) VS.  
CORROSION TIME FOR SAMPLE  
NO. IF-2

D 52003



CRYSTAL No 2 SURFACE FILM CONDITION AS ELECTROPOLISHED

	@ 344°	@ 254°	@ 164°	@ 74°
M1	44.2	41.2	43.9	41.2
R1	45.8	45.2	46.2	45.2
M2	44.2	41.1	44.0	41.2
R2	46.0	45.1	45.9	45.1
AV	44.20	41.15	43.95	41.20
	45.90	45.15	46.05	45.15

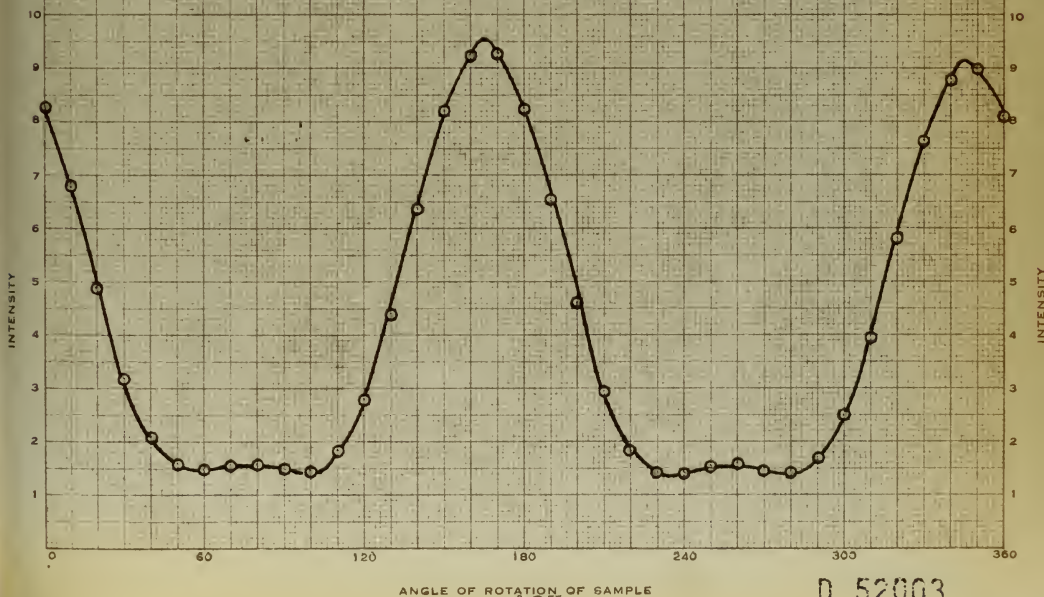
M1	44.20	R1	45.90
M2	41.15	R2	45.15
$\Delta M_{12}$	3.05	$\Delta R_{12}$	0.75

M3	43.95	R3	46.05
M4	41.20	R4	45.15
$\Delta M_{34}$	2.75	$\Delta R_{34}$	0.90

AV  $\Delta M$  2.30 PHASE SHIFT  
AV  $\Delta R$  0.83 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE No 17

240 PPM  $N_2$ 

DATE 12 MARCH 1962 AM

CRYSTAL No 2

SURFACE FILM CONDITION

1st AUTOCALIB 15 MIN. @ 27°C

@ ... 276 ...		@ ... 186 ...		@ ... 096 ...		@ ... 006 ...	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>1</sub>	M <sub>4</sub>	R <sub>4</sub>
44.1	45.8	40.0	45.2	43.7	45.8	40.2	45.3
43.7	45.6	40.1	45.3	43.7	45.8	40.2	45.4
43.3	45.8						
Av 43.90	45.73	40.05	45.25	43.70	45.8	40.2	45.35

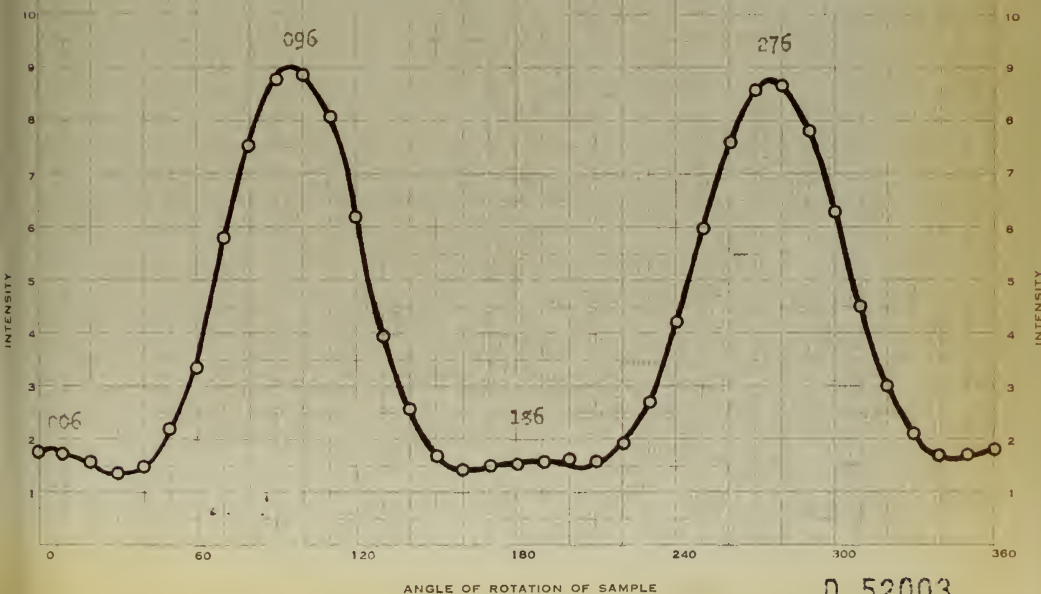
M <sub>1</sub> 43.90	R <sub>1</sub> 45.73
M <sub>2</sub> 40.05	R <sub>2</sub> 45.25
$\Delta M_{12}$ 3.85	$\Delta R_{12}$ 0.48

M <sub>3</sub> 43.70	R <sub>3</sub> 45.80
M <sub>4</sub> 40.20	R <sub>4</sub> 45.35
$\Delta M_{34}$ 3.50	$\Delta R_{34}$ 0.45

Av  $\Delta M$  3.67 PHASE SHIFTAv  $\Delta R$  0.46 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 17

240 PPM  $\text{Mg}$ 

DATE 14 MARCH 1952

CRYSTAL NO .2

SURFACE FILM CONDITION

2nd AUTOCLAVE 15 MIN. at 233°C

@ 275		@ 175		@ 095		@ 005	
M1	R1	M2	R2	M3	R3	M4	R4
45.2	45.8	41.2	45.3	45.1	45.7	41.2	45.2
45.2	45.7	41.4	45.3	45.1	45.7	41.1	45.2
AV. 45.2	45.75	41.30	45.30	45.10	45.70	41.15	45.20

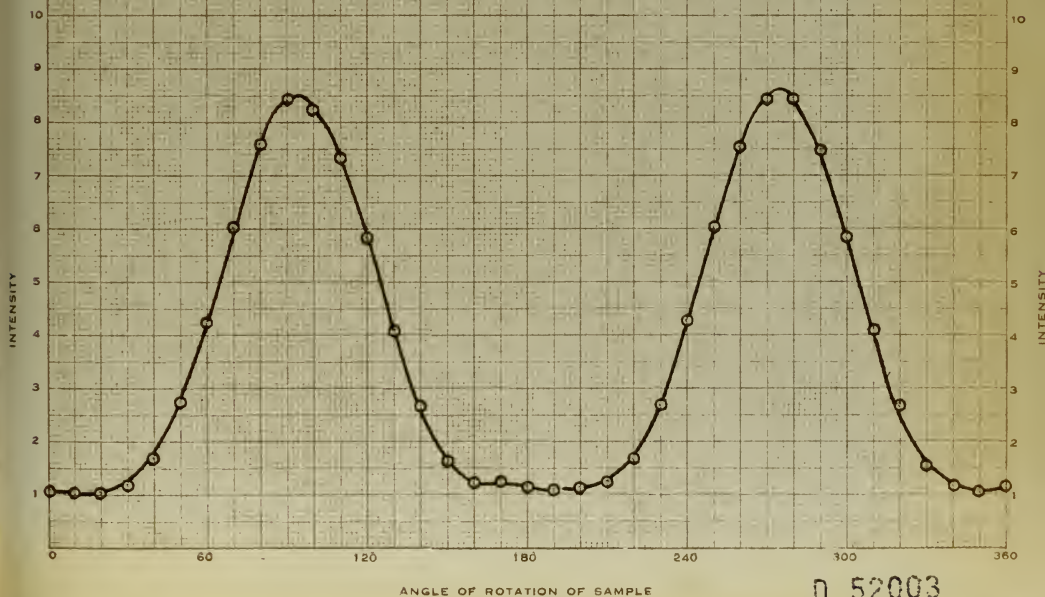
M <sub>1</sub>	45.20	R <sub>1</sub>	45.75
M <sub>2</sub>	41.30	R <sub>2</sub>	45.30
$\Delta M_{12}$	3.90	$\Delta R_{12}$	0.45

M <sub>3</sub>	45.10	R <sub>3</sub>	45.70
M <sub>4</sub>	41.15	R <sub>4</sub>	45.20
$\Delta M_{34}$	3.95	$\Delta R_{34}$	0.50

AV  $\Delta M$  3.92 PHASE SHIFTAV  $\Delta R$  0.47 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CRYSTAL NO. 2

SURFACE FILM CONDITION

3rd AUTOCLAVE 15 MIN. at 233°C

@ 242		@ 152		@ 062		@ 332	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.1	45.8	41.0	45.30	45.0	45.8	40.7	45.4
45.2	45.8	40.8	45.40	45.1	45.8	40.7	45.4
AV 45.15	45.80	40.90	45.35	45.05	45.80	40.70	45.40

M <sub>1</sub>	45.15	R <sub>1</sub>	45.80
M <sub>2</sub>	40.90	R <sub>2</sub>	45.35
$\Delta M_{12}$	4.25	$\Delta R_{12}$	0.45

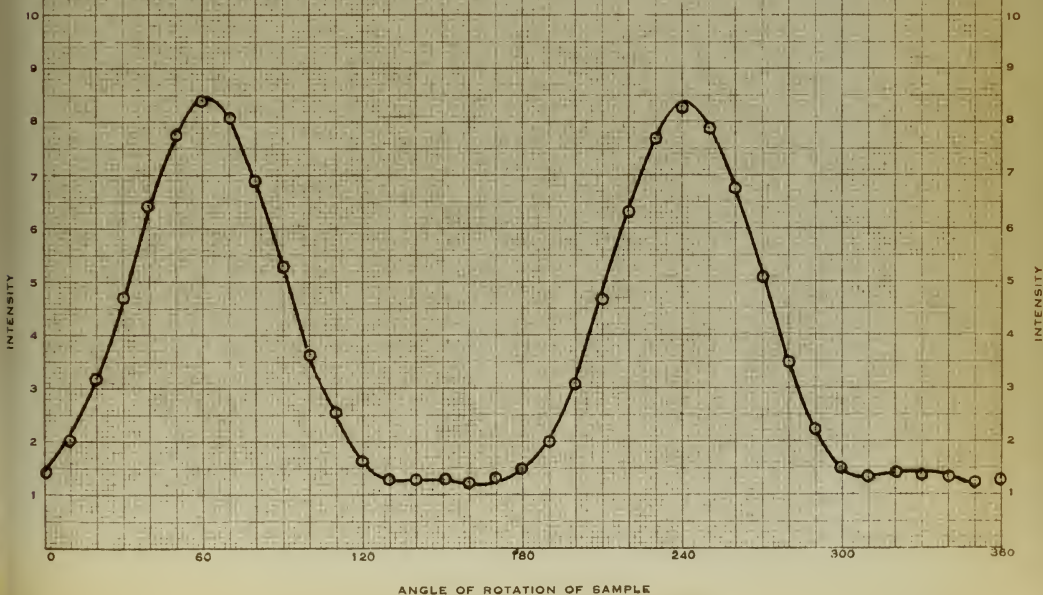
M <sub>3</sub>	45.05	R <sub>3</sub>	45.80
M <sub>4</sub>	40.70	R <sub>4</sub>	45.40
$\Delta M_{34}$	4.35	$\Delta R_{34}$	0.40

AV  $\Delta M$  4.30 PHASE SHIFT

AV  $\Delta R$  0.42 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







	@ 011		@ 281		@ 191		@ 101	
	M1	RT	M2	R2	M3	R3	M4	R4
	44.6	46.1	40.1	45.7	44.5	45.2	40.0	45.7
	44.6	46.1	40.0	45.7	44.4	45.9	39.8	45.7
AV	44.60	46.20	40.05	45.70	44.45	45.90	39.90	45.70

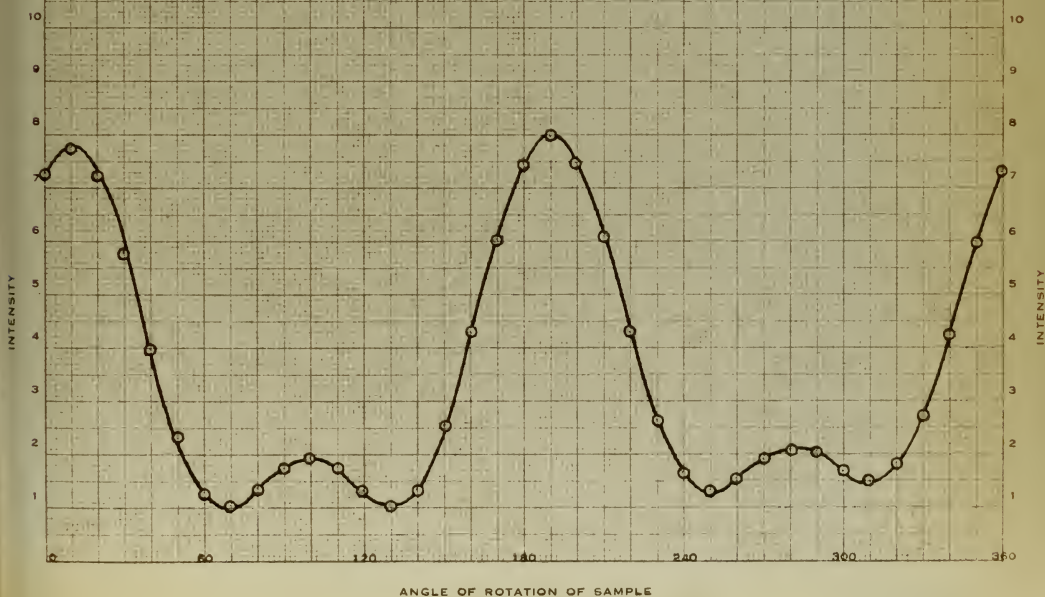
M <sub>1</sub>	44.60	R <sub>1</sub>	46.10
M <sub>2</sub>	40.05	R <sub>2</sub>	45.70
$\Delta M_{12}$	4.55	$\Delta R_{12}$	0.40

M <sub>3</sub>	44.45	R <sub>3</sub>	45.90
M <sub>4</sub>	39.90	R <sub>4</sub>	45.70
$\Delta M_{34}$	4.55	$\Delta R_{34}$	0.20

AV  $\Delta M$  4.55 PHASE SHIFTAV  $\Delta R$  0.30 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







	@ 237		@ 147		@ 957		@ 327	
	M1	R1	M2	R2	M3	R3	M4	R4
	43.3	45.7	38.8	45.4	43.4	45.6	38.7	45.7
	43.4	45.6	38.9	45.6	43.2	45.7	38.7	45.5
AV	43.35	45.65	38.85	45.50	43.30	45.65	38.70	45.60

M<sub>1</sub> 43.35      R<sub>1</sub> 45.65  
 M<sub>2</sub> 38.85      R<sub>2</sub> 45.50  
 ΔM<sub>12</sub> 4.50      ΔR<sub>12</sub> 0.15

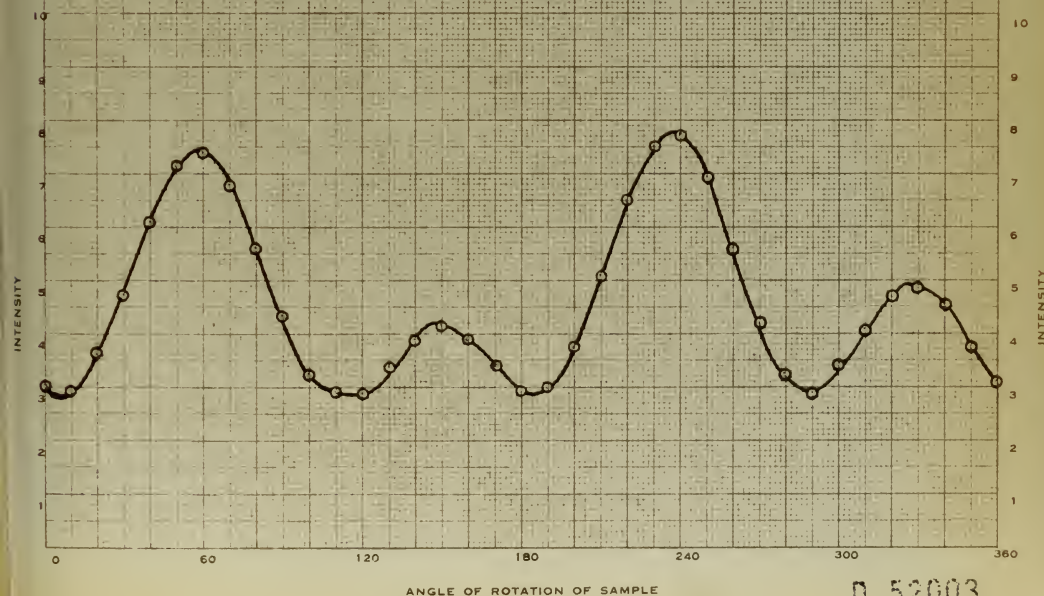
M<sub>3</sub> 43.30      R<sub>3</sub> 45.65  
 M<sub>4</sub> 38.70      R<sub>4</sub> 45.60  
 ΔM<sub>34</sub> 4.60      ΔR<sub>34</sub> 0.05

AV ΔM 4.55      PHASE SHIFT

AV ΔR 0.10      PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



CRYSTAL No 2

SURFACE FILM CONDITION

5th AUTOCLAVE 15 MIN. @ 233°C

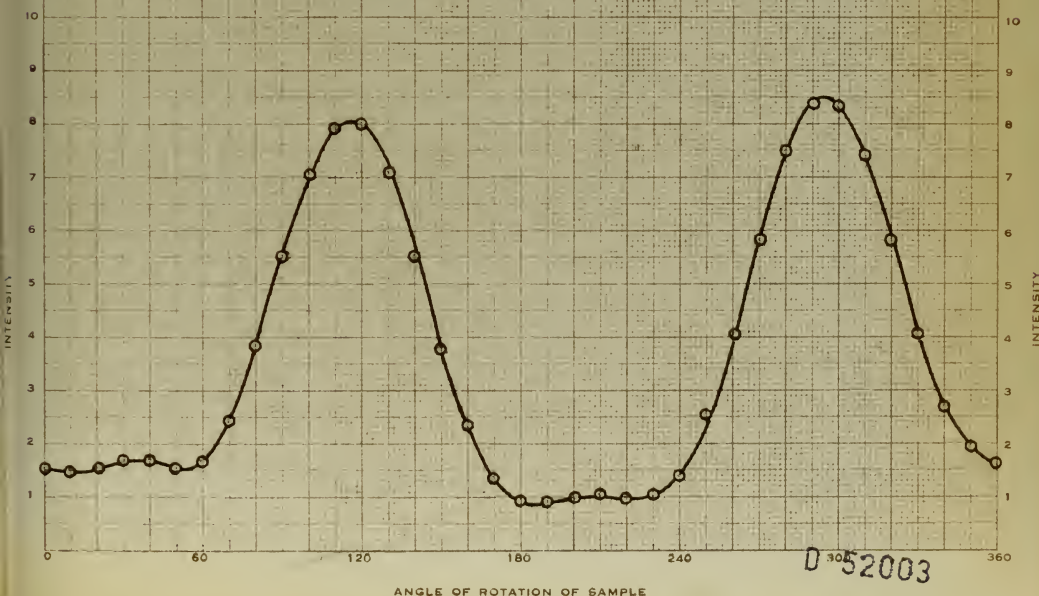
@ 295		@ 205		@ 115		@ 025	
M <sub>1</sub>	R	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.0	45.6	40.8	45.4	45.1	45.7	40.6	45.6
46.2	45.5	40.9	45.5	45.1	45.6	40.5	45.6
AV. 46.10	45.55	40.85	45.45	45.10	45.65	40.55	45.60

M <sub>1</sub>	46.10	R <sub>1</sub>	45.55
M <sub>2</sub>	40.85	R <sub>2</sub>	45.45
$\Delta M_{12}$	5.25	$\Delta R_{12}$	0.10

M <sub>3</sub>	45.10	R <sub>3</sub>	45.65
M <sub>4</sub>	40.55	R <sub>4</sub>	45.60
$\Delta M_{34}$	4.55	$\Delta R_{34}$	0.05

AV  $\Delta M$  4.92 PHASE SHIFT  
 AV  $\Delta R$  0.07 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





CRYSTAL NO 2

SURFACE FILM CONDITION

7:15 AMT. GRAVE 12 MAY. 1953

@ 224		@ 154		@ 044		@ 314	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.5	45.6	40.9	45.4	45.7	45.6	40.5	45.9
45.7	45.6	40.7	45.4	45.7	45.6	40.8	45.9
AV 45.60	45.60	40.80	45.40	45.70	45.60	40.50	45.40

M <sub>1</sub> 45.60	R <sub>1</sub> 45.60
M <sub>2</sub> 40.80	R <sub>2</sub> 45.40
$\Delta M_{12}$ 4.80	$\Delta R_{12}$ 0.20

M <sub>3</sub> 45.70	R <sub>3</sub> 45.60
M <sub>4</sub> 40.50	R <sub>4</sub> 45.40
$\Delta M_{34}$ 5.20	$\Delta R_{34}$ 0.20

AV  $\Delta M$  5.00

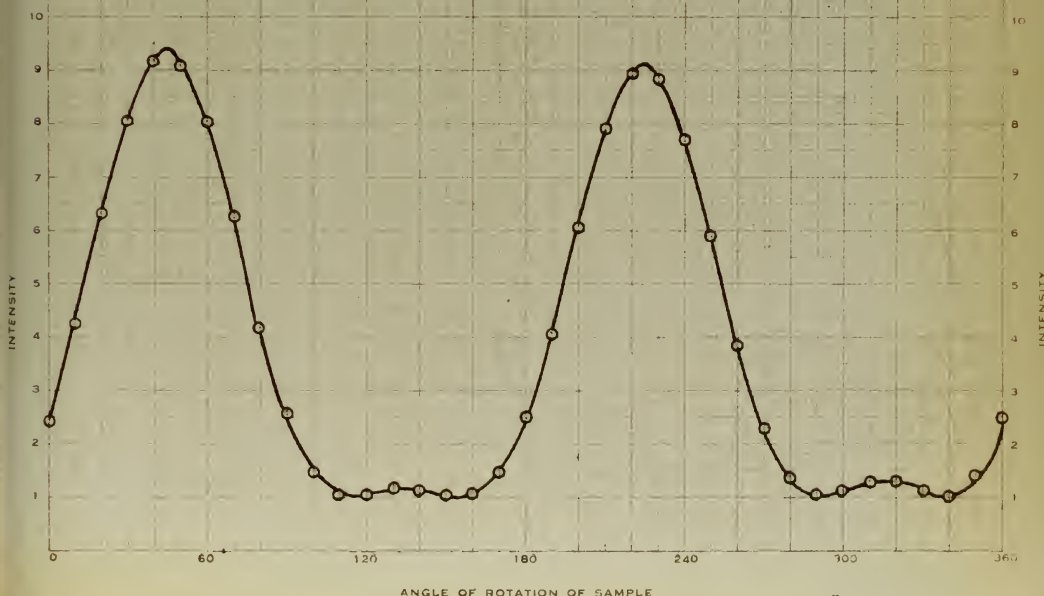
PHASE SHIFT

AV  $\Delta R$  0.20

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE No 1F

PPM 11

DATE 27 MARCH 1957 AM

CRYSTAL No 2

SURFACE FILM CONDITION

2% AUTOCLAVE 15 MIN. @ 235°C

@ 399		@ 349		@ 129		@ 939	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.2	45.6	41.4	45.4	46.4	45.6	40.9	45.6
46.4	45.7	41.4	45.4	46.2	45.6	40.7	45.5
Av 46.30	45.65	41.40	45.40	46.30	45.60	40.80	45.55

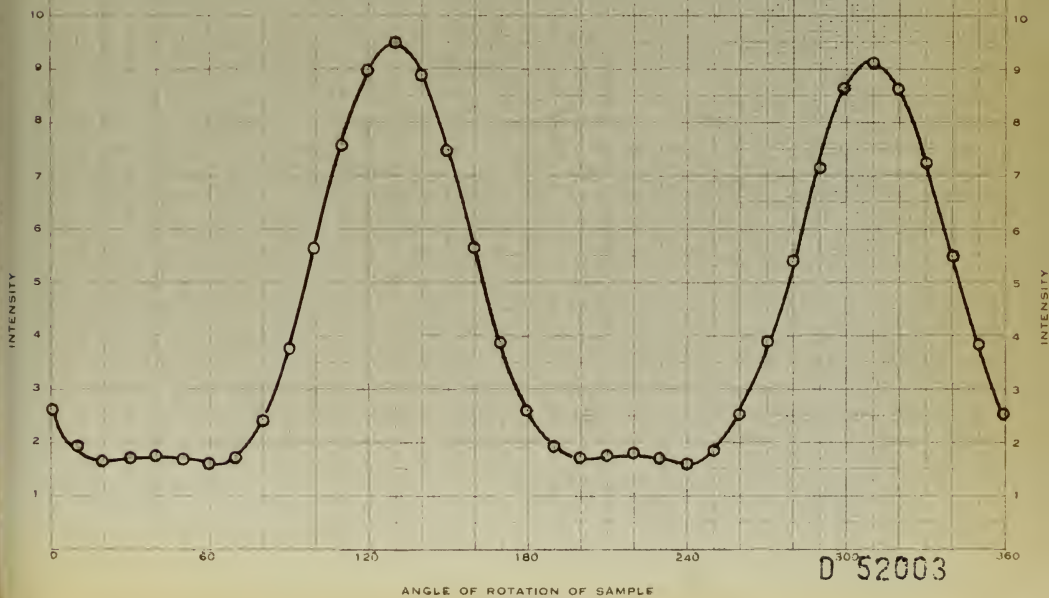
M <sub>1</sub>	46.30	R <sub>1</sub>	45.65
M <sub>2</sub>	41.40	R <sub>2</sub>	45.40
$\Delta M_{12}$	4.90	$\Delta R_{12}$	0.25

M <sub>3</sub>	46.30	R <sub>3</sub>	45.60
M <sub>4</sub>	40.80	R <sub>4</sub>	45.55
$\Delta M_{34}$	5.50	$\Delta R_{34}$	0.05

Av  $\Delta M$  5.20 PHASE SHIFTAv  $\Delta R$  0.15 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



CRYSTAL No. 2 SURFACE FILM COMPOSITION 9th AUTOCLAVE 15 MIN @ 233°C

@ 298		@ 208		@ 118		@ 028	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
47.2	45.7	41.6	45.5	46.9	45.7	41.5	45.3
47.0	45.7	41.7	45.5	46.8	45.6	41.5	45.4
Av. 47.10		41.65		46.85		41.50	

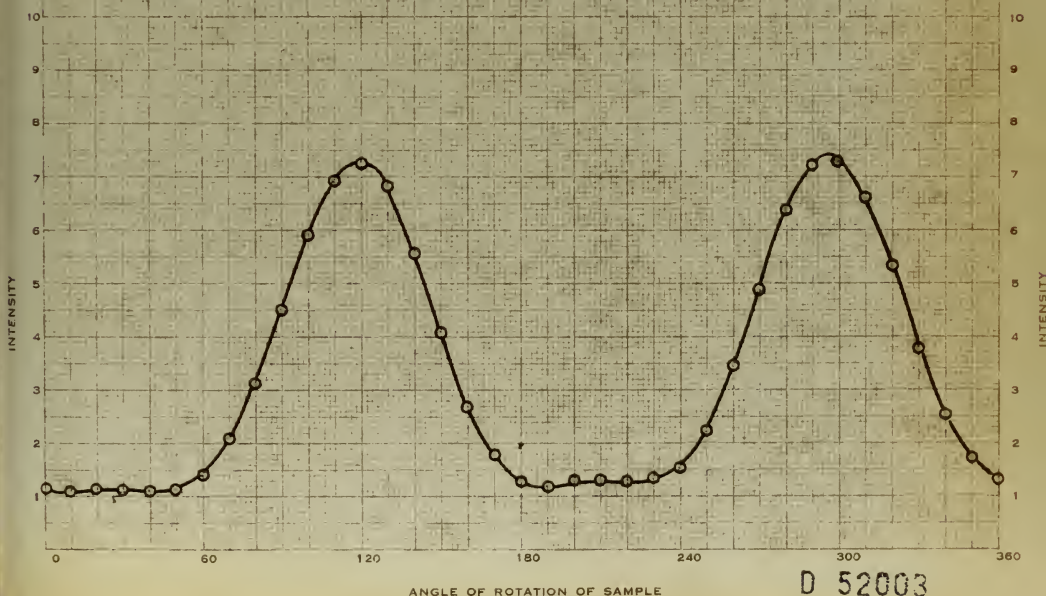
M <sub>1</sub>	47.10	R <sub>1</sub>	45.70
M <sub>2</sub>	41.65	R <sub>2</sub>	45.50
$\Delta M_{12}$	5.45	$\Delta R_{12}$	0.20

M <sub>3</sub>	46.85	R <sub>3</sub>	45.65
M <sub>4</sub>	41.50	R <sub>4</sub>	45.35
$\Delta M_{34}$	5.35	$\Delta R_{34}$	0.30

Av  $\Delta M$  5.40 PHASE SHIFT  
Av  $\Delta R$  0.25 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE No 1F

240 PPM  $N_2$ 

DATE 31 MARCH 1952 AM

CRYSTAL No 2

SURFACE FILM CONDITION

10th AUTOCLAVE 15 MIN. @ 233°C

@ 114		@ 224		@ 174		@ 044	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.9	45.8	41.6	45.4	46.6	45.7	41.6	45.7
46.7	45.8	41.7	45.6	46.9	45.8	41.6	45.5
AV 46.80	45.80	41.65	45.50	46.75	45.75	41.60	45.60

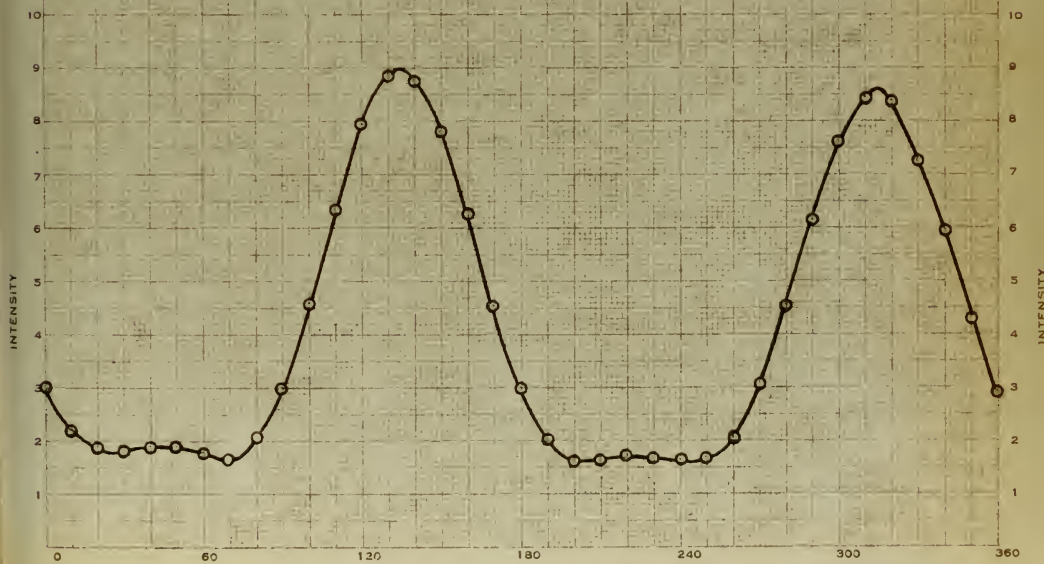
M <sub>1</sub>	46.80	R <sub>1</sub>	45.80
M <sub>2</sub>	41.65	R <sub>2</sub>	45.50
$\Delta M_{12}$	5.15	$\Delta R_{12}$	0.30

M <sub>3</sub>	46.75	R <sub>3</sub>	45.75
M <sub>4</sub>	41.60	R <sub>4</sub>	45.60
$\Delta M_{34}$	5.15	$\Delta R_{34}$	0.15

AV  $\Delta M$  5.15 PHASE SHIFTAV  $\Delta R$  0.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





CRYSTAL NO. 2

SURFACE FILM CONDITION

11th AUTOCLAVE 15 MIN @ 233°C

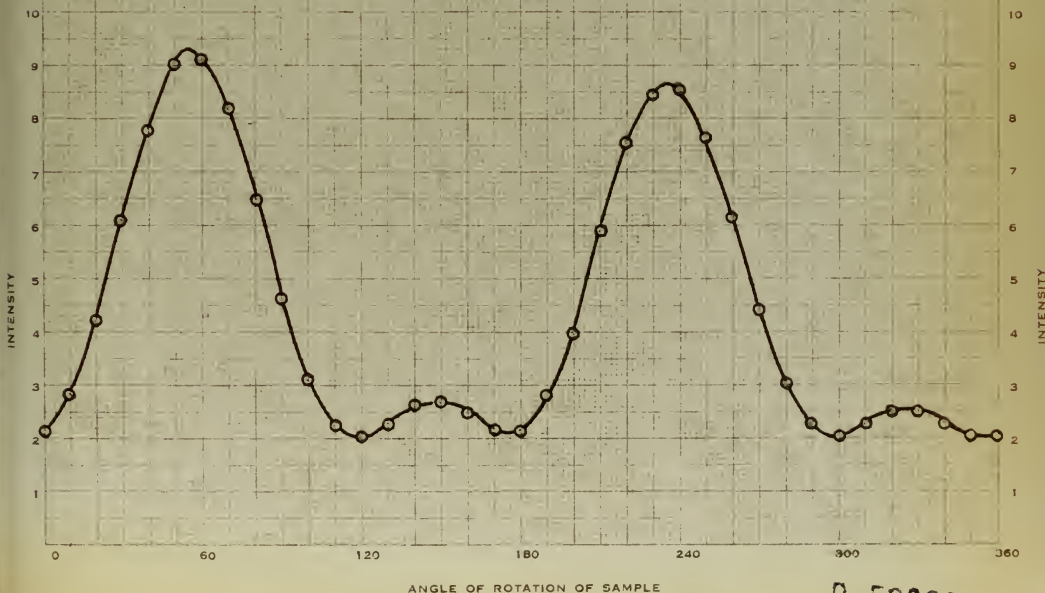
@ 236		@ 146		@ 056		@ 326	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.5	45.8	39.9	45.7	44.8	45.9	39.6	45.6
44.6	45.8	39.6	45.7	44.7	45.7	39.6	45.7
AV. 44.55	45.80	39.75	45.70	44.75	45.80	39.60	45.65

M <sub>1</sub>	44.55	R <sub>1</sub>	45.80
M <sub>2</sub>	39.75	R <sub>2</sub>	45.70
$\Delta M_{12}$	4.80	$\Delta R_{12}$	0.10

M <sub>3</sub>	44.75	R <sub>3</sub>	45.80
M <sub>4</sub>	39.60	R <sub>4</sub>	45.65
$\Delta M_{34}$	5.15	$\Delta R_{34}$	0.15

AV  $\Delta M$  4.97 PHASE SHIFT  
 AV  $\Delta R$  0.12 PHASE ROTATION  
 PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE No. 1F

240 PPM N<sub>2</sub>

DATE 4 APR 11 1952 AN

CRYSTAL No. 2

SURFACE FILM CONDITION

13th AUTOCLAVE 15 MIN. @ 233°C

@ 300		@ 210		@ 120		@ 030	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.1	45.9	39.8	45.7	45.0	45.9	39.4	45.7
44.6	45.8	39.6	45.7	44.7	45.7	39.4	45.5
Av. 44.50	45.85	39.70	45.70	44.85	45.80	39.40	45.60

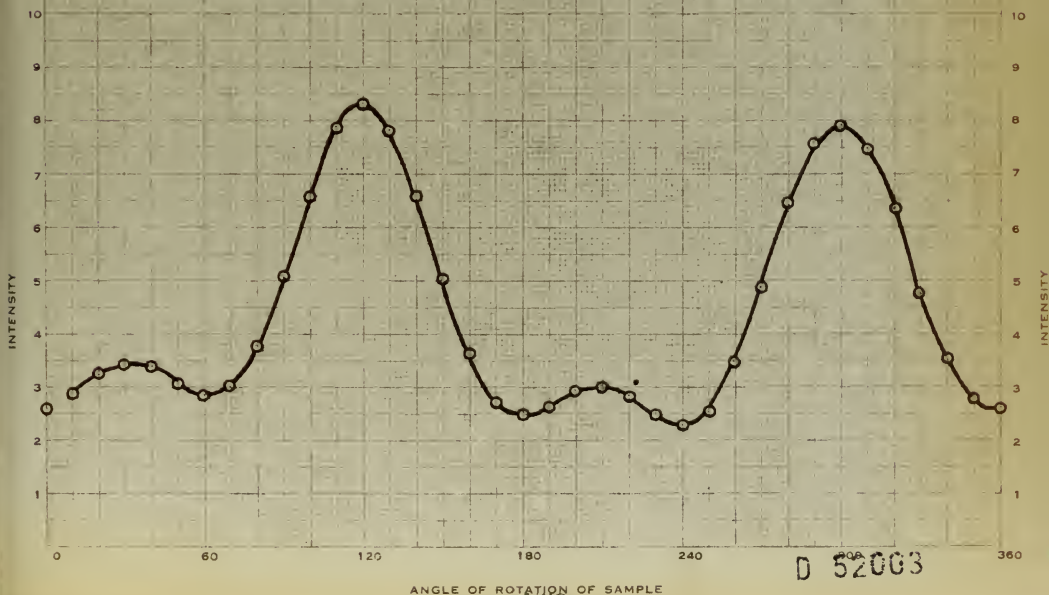
M <sub>1</sub> 44.50	R <sub>1</sub> 45.85
M <sub>2</sub> 39.70	R <sub>2</sub> 45.70
$\Delta M_{12}$ 4.80	$\Delta R_{12}$ 0.15

M <sub>3</sub> 44.85	R <sub>3</sub> 45.80
M <sub>4</sub> 39.40	R <sub>4</sub> 45.50
$\Delta M_{34}$ 5.45	$\Delta R_{34}$ .20

Av  $\Delta M$  5.07 PHASE SHIFT  
 Av  $\Delta R$  0.17 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE No 1F 240 PPM N<sub>2</sub>

DATE 5 APRIL 1952 AX

CRYSTAL No 2

SURFACE FILM CONDITION

15th AUTOCLAVE OF 15 MIN. @ 233°C

@ 355

@ 265

@ 175

@ 085

M<sub>1</sub>R<sub>1</sub>M<sub>2</sub>R<sub>2</sub>M<sub>3</sub>R<sub>3</sub>M<sub>4</sub>R<sub>4</sub>

48.3

45.8

40.8

45.9

47.9

45.9

41.4

45.7

48.1

45.9

41.0

45.9

47.8

45.9

41.5

45.8

AV

48.20

45.85

40.90

45.90

47.85

45.90

41.45

45.75

M<sub>1</sub> 48.20R<sub>1</sub> 45.85M<sub>2</sub> 40.90R<sub>2</sub> 45.90 $\Delta M_{12}$  7.30 $\Delta R_{12}$  .05M<sub>3</sub> 47.85R<sub>3</sub> 45.90M<sub>4</sub> 41.45R<sub>4</sub> 45.75 $\Delta M_{34}$  6.40 $\Delta R_{34}$  .15AV  $\Delta M$  6.85

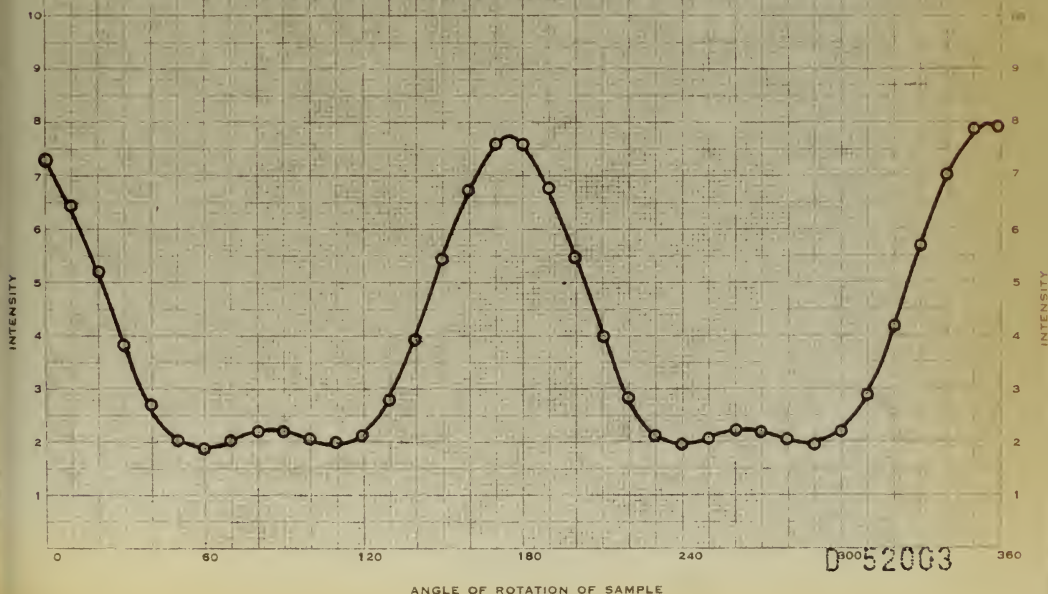
PHASE SHIFT

AV  $\Delta R$  0.05

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE





CONFIDENTIAL

Run #2

Corrosion film was grown at 295° C

D 52003

CONFIDENTIAL



CRYSTAL NO. 1

SURFACE FILM CONDITION

21. 11/11/50

@ 0°		@ 078		@ 157		@ 235	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.6	46.2	38.7	44.6	45.1	46.5	38.7	44.5
45.5	46.3	39.0	44.5	45.0	46.4	38.7	44.5
AV 45.55	46.25	38.85	44.55	45.05	46.45	38.70	44.50

M <sub>1</sub>	45.55	R <sub>1</sub>	46.25
M <sub>2</sub>	38.85	R <sub>2</sub>	44.55
$\Delta M_2$	6.70	$\Delta R_2$	1.70

M <sub>1</sub>	45.05	R <sub>1</sub>	46.45
M <sub>2</sub>	38.70	R <sub>2</sub>	44.50
$\Delta M_{34}$	6.35	$\Delta R_{34}$	1.95

AV  $\Delta M$  6.53

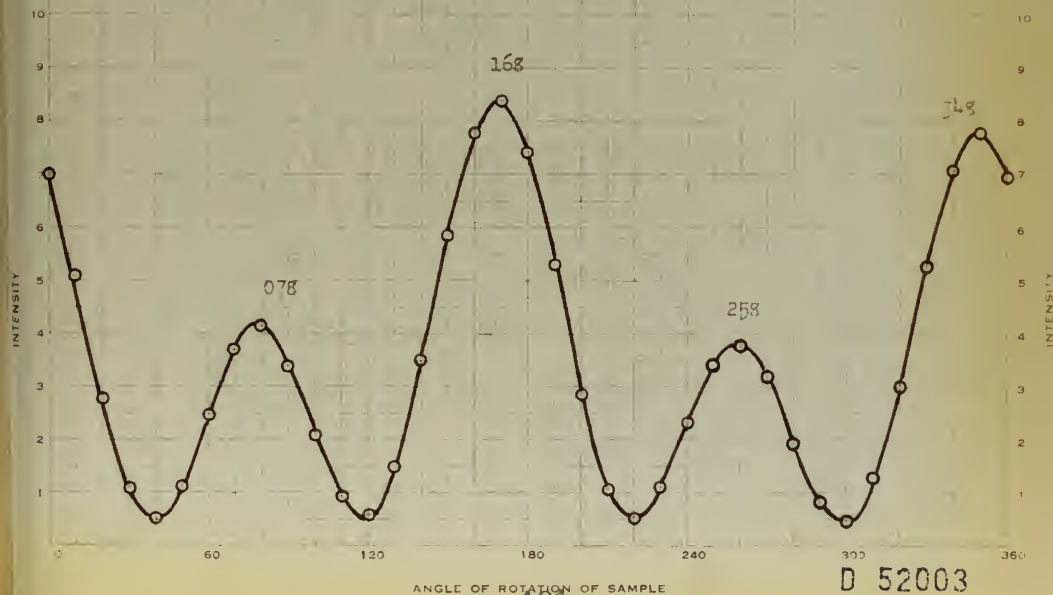
PHASE SHIFT

AV  $\Delta R$  1.83

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





CRYSTAL No. 1

SURFACE FILM CONDITION 1st AUTOCLAVE OF 15 MIN. @ 295°C

@ 064

@ 154

@ 244

@ 334

M<sub>1</sub>R<sub>1</sub>M<sub>2</sub>R<sub>2</sub>M<sub>3</sub>R<sub>3</sub>M<sub>4</sub>R<sub>4</sub>

51.4

44.9

33.6

45.6

50.7

44.9

32.7

46.1

51.3

44.3

33.6

45.7

50.7

44.7

32.7

46.0

Av.

51.35

44.20

33.60

45.65

50.70

44.80

32.70

46.05

M<sub>1</sub>

51.35

R<sub>1</sub>

44.20

M<sub>2</sub>

33.60

R<sub>2</sub>

45.65

 $\Delta M_{12}$ 

17.75

 $\Delta R_{12}$ 

- .75

M<sub>3</sub>

50.70

R<sub>3</sub>

44.80

M<sub>4</sub>

32.70

R<sub>4</sub>

46.05

 $\Delta M_{34}$ 

18.00

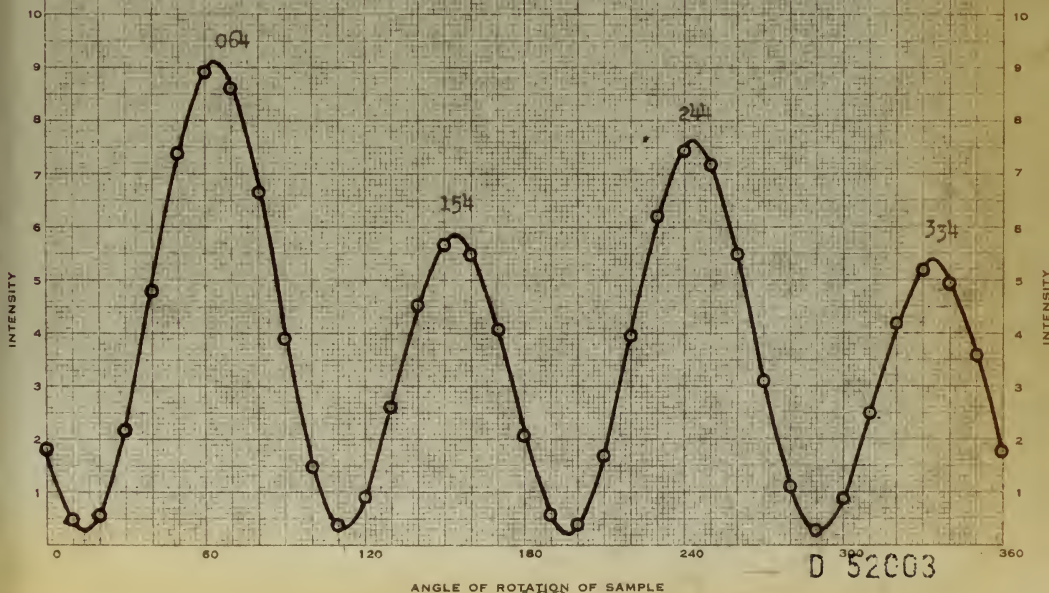
 $\Delta R_{34}$ 

-1.25

Av  $\Delta M$  17.88 PHASE SHIFTAv  $\Delta R$  -1.00 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL NO 2

SURFACE FILM CONDITION

2nd ELECTROPOLISH

@ 060		@ 060		@ 148.5		@ 239	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
41.1	45.0	42.8	45.7	40.8	45.0	42.6	45.7
41.1	45.0	42.8	45.7	41.0	45.0	42.4	45.6
AV 41.10	45.00	42.80	45.70	40.90	45.00	42.50	45.65

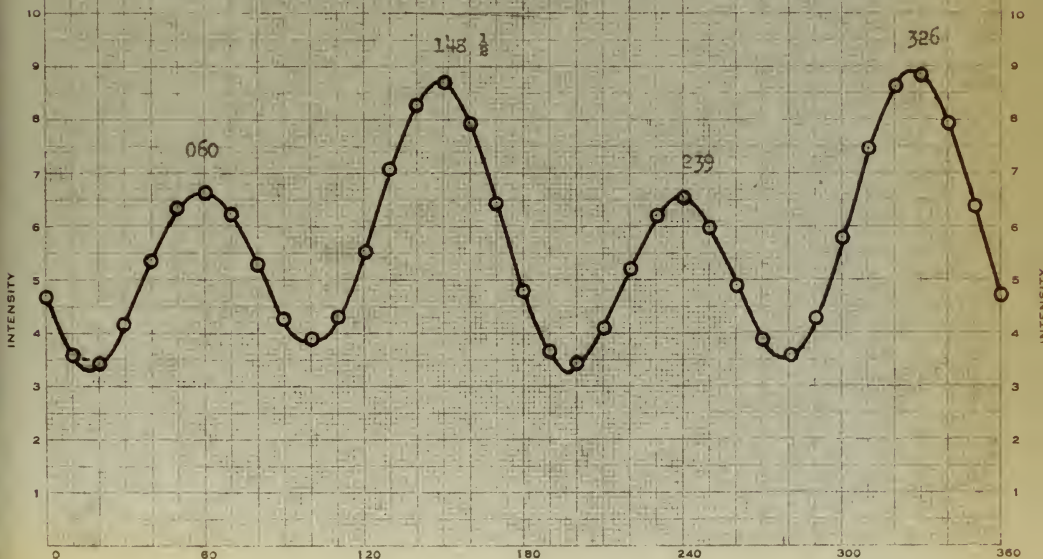
M <sub>1</sub>	41.10	R <sub>1</sub>	45.00
M <sub>2</sub>	42.80	R <sub>2</sub>	45.70
$\Delta M_{12}$	-1.70	$\Delta R_{12}$	-.70

M <sub>3</sub>	40.90	R <sub>3</sub>	45.00
M <sub>4</sub>	42.50	R <sub>4</sub>	45.65
$\Delta M_{34}$	-1.60	$\Delta R_{34}$	-.65

AV  $\Delta M$  -1.65 PHASE SHIFTAV  $\Delta R$  -.68 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO. 2A

6 PPM  $N_2$ 

DATE 15 APRIL 1952

CRYSTAL NO. 2

SURFACE FILM CONDITION 1st AUTOCLAVE OF 15 MIN. @ 295°C

@ 052		@ 052		@ 140		@ 230	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
44.6	50.1	42.3	40.9	44.2	50.1	42.1	40.6
44.5	44.9	42.2	41.1	44.2	50.3	41.7	40.4
AV. 44.60		42.25		44.20		41.90	
50.00		41.00		50.20		40.50	

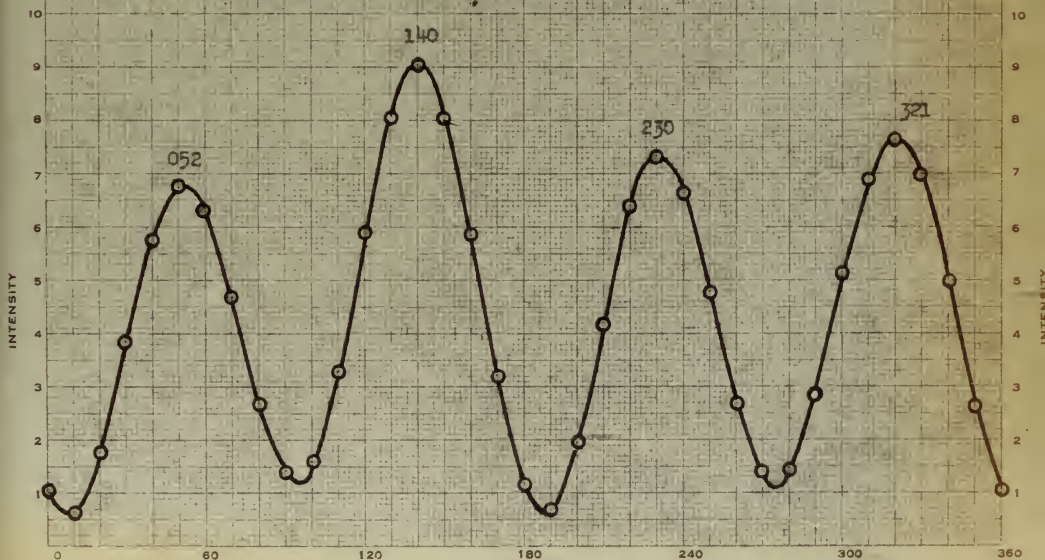
M <sub>1</sub> 44.60	R <sub>1</sub> 50.00
M <sub>2</sub> 42.25	R <sub>2</sub> 41.00
$\Delta M_{12}$ 2.35	$\Delta R_{12}$ 9.00

M <sub>3</sub> 44.20	R <sub>3</sub> 50.20
M <sub>4</sub> 41.90	R <sub>4</sub> 40.50
$\Delta M_{34}$ 2.30	$\Delta R_{34}$ 9.70

AV  $\Delta M$  2.33 PHASE SHIFT  
 AV  $\Delta R$  9.35 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







CRYSTAL NO 2

SURFACE FILM COMPOSITION

3rd TEST 101122

① @ 23		② @ 150		③ @ 150		④ @ 242	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
41.1	45.0	42.7	45.7	40.5	44.8	42.1	45.6
41.0	45.0	42.5	45.7	40.7	45.0	42.3	45.6
AV 41.05	45.00	42.60	45.70	40.60	44.90	42.35	45.60

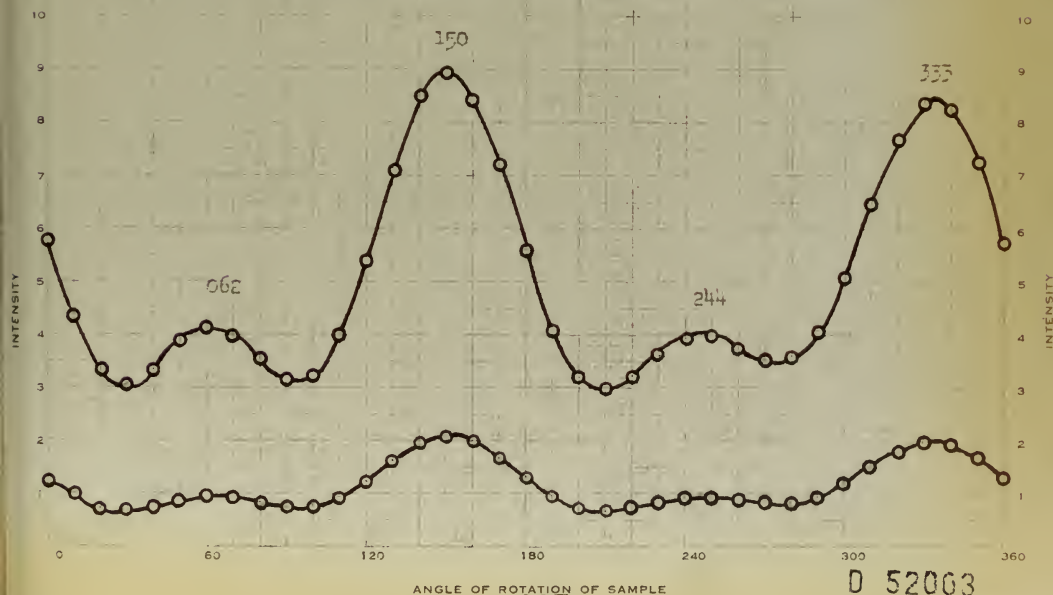
M	41.05	R	45.00
M <sub>1</sub>	42.60	R <sub>2</sub>	45.70
$\Delta M_1$	-1.55	$\Delta R_2$	-.70

M <sub>2</sub>	40.60	R <sub>3</sub>	44.90
M <sub>3</sub>	42.35	R <sub>4</sub>	45.60
$\Delta M_{24}$	-1.75	$\Delta R_{31}$	-.70

AV  $\Delta M$  -1.65 PHASE SHIFTAV  $\Delta R$  -.70 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE NO. 3

PPM 71

DATE 11 MAR 61 1057 13

CRYSTAL NO. 3

SURFACE FILM CONDITION

PL 1122 111111

@ 307		@ 127		@ 127		@ 037	
M1	R1	M2	R2	M3	R3	M4	R4
46.0	46.7	39.6	45.1	46.0	46.7	39.6	45.0
46.0	46.7	39.4	45.1	46.0	46.7	39.6	45.0
AV 46.00	46.70	39.50	45.10	46.00	46.70	39.60	45.00

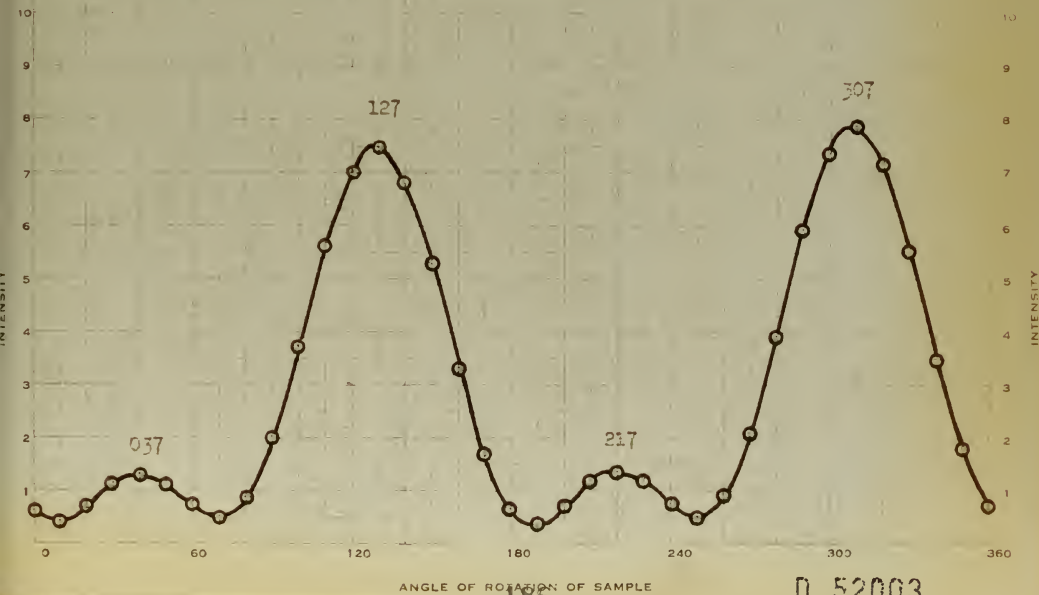
M <sub>1</sub> 46.00	R <sub>1</sub> 46.70
M <sub>2</sub> 39.50	R <sub>2</sub> 45.10
$\Delta M_{12}$ 6.50	$\Delta R_{12}$ 1.60

M <sub>3</sub> 46.00	R <sub>3</sub> 46.70
M <sub>4</sub> 39.60	R <sub>4</sub> 45.00
$\Delta M_{34}$ 6.40	$\Delta R_{34}$ 1.70

AV  $\Delta M$  6.45 PHASE SHIFTAV  $\Delta R$  1.65 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE NO 2P

70 PPM  $N_2$ 

DATE 13 APRIL 1962 PM

CRYSTAL NO 3

SURFACE FILM CONDITION 1st AUTOCLAVE 15 MIN. @ 255°C

@ 063		@ 153		@ 243		@ 333	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
51.4	45.5	37.0	46.4	52.0	45.1	36.3	46.6
51.8	45.4	36.5	46.4	51.8	44.9	37.0	46.4
AV. 51.60	45.45	36.75	46.40	51.90	45.00	36.90	46.50

M <sub>1</sub>	51.60	R <sub>1</sub>	45.45
M <sub>2</sub>	36.75	R <sub>2</sub>	46.40
$\Delta M_{12}$	14.85	$\Delta R_{12}$	-0.95

M <sub>3</sub>	51.90	R <sub>3</sub>	45.00
M <sub>4</sub>	36.90	R <sub>4</sub>	46.50
$\Delta M_{34}$	15.00	$\Delta R_{34}$	-1.50

AV  $\Delta M$  14.92 PHASE SHIFTAV  $\Delta R$  -1.22 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE NO 20

113 PPM

DATE 12 APRIL 1962 AM

CRYSTAL NO. 1

SURFACE FILM CONDITION

SIL ELECTRO POLISH

@ 270		@ 001		@ 090		@ 180	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.3	46.6	40.2	44.7	45.1	46.6	40.5	45.0
45.5	46.5	40.7	44.9	45.1	46.5	40.2	45.1
AV. 45.40	46.55	40.75	44.80	45.10	46.55	40.35	45.03

M <sub>1</sub> 45.40	R <sub>1</sub> 46.55
M <sub>2</sub> 40.75	R <sub>2</sub> 44.80
$\Delta M_{12}$ 4.65	$\Delta R_{12}$ 1.75

M <sub>3</sub> 45.10	R <sub>3</sub> 46.55
M <sub>4</sub> 40.35	R <sub>4</sub> 45.03
$\Delta M_{34}$ 4.75	$\Delta R_{34}$ 1.50

AV  $\Delta M$  4.70

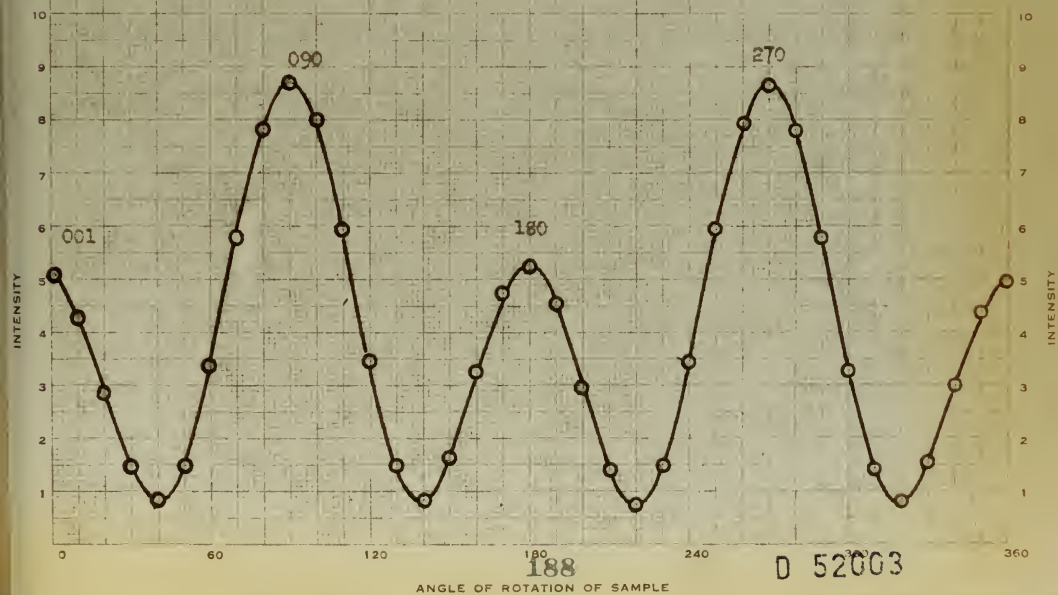
PHASE SHIFT

AV  $\Delta R$  1.62

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE

188  
ANGLE OF ROTATION OF SAMPLE

D 52003





SAMPLE No. 20

113ppm K<sub>2</sub>

DATE 16 APRIL 1952 PM

CRYSTAL No. 3

SURFACE FILM CONDITION

1st AUTOCLAVE OF 15 MIN. @ 295°C

@ 009		@ 098		@ 188 $\frac{1}{2}$		@ 278 $\frac{1}{2}$	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
34.4	46.9	50.3	44.6	33.8	46.9	50.5	44.5
34.0	47.0	50.0	44.5	33.7	46.8	50.4	44.6
AV. 34.20	46.95	50.15	44.60	33.75	46.85	50.45	44.55

M <sub>1</sub> 34.20	R <sub>1</sub> 46.95
M <sub>2</sub> 50.15	R <sub>2</sub> 44.60
$\Delta M_{12}$ -15.95	$\Delta R_{12}$ 2.35

M <sub>3</sub> 33.75	R <sub>3</sub> 46.85
M <sub>4</sub> 50.45	R <sub>4</sub> 44.55
$\Delta M_{34}$ -16.70	$\Delta R_{34}$ 2.30

AV  $\Delta M$  -16.33

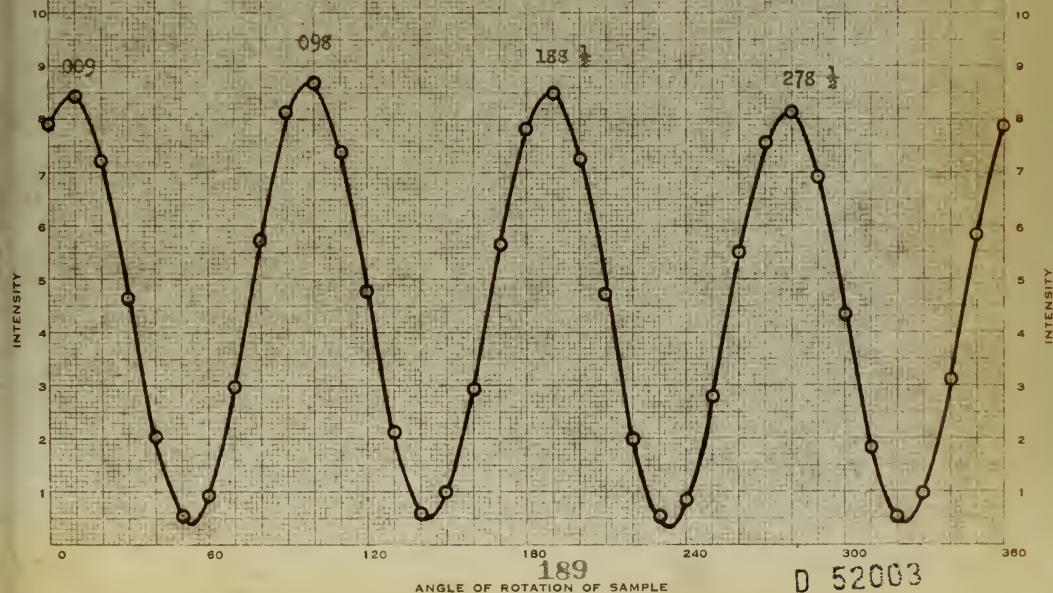
PHASE SHIFT

AV  $\Delta R$  2.33

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003



SAMPLE No. 2D

147 PPM N<sub>2</sub>

DATE 10 APRIL 1952 PM

CRYSTAL No. 1

SURFACE FILM CONDITION 2nd ELECTROFOLISH

@ 227		@ 257		@ 257		@ 267	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
45.8	46.4	39.2	44.6	45.9	46.5	39.0	44.6
45.8	46.5	39.3	44.6	45.9	46.5	39.0	44.6
AV. 45.80	46.45	39.25	44.60	45.90	46.50	39.00	44.60

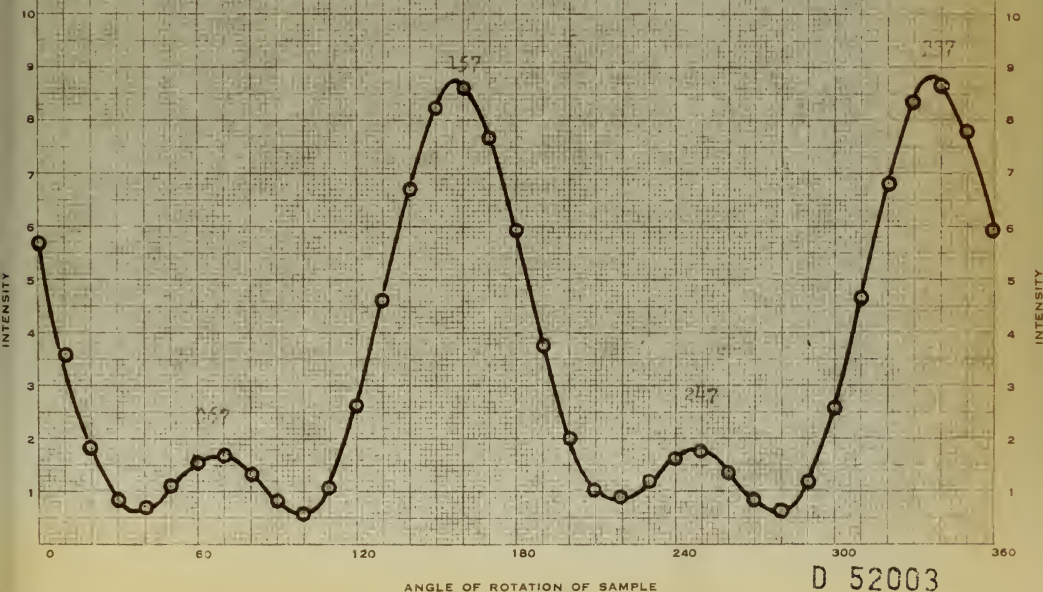
M <sub>1</sub>	45.80	R	46.45
M <sub>2</sub>	39.25	R	44.60
$\Delta M_{12}$	6.55	$\Delta R_{12}$	1.85

M <sub>3</sub>	45.90	R	46.50
M <sub>4</sub>	39.00	R	44.60
$\Delta M_{34}$	6.90	$\Delta R_{34}$	1.90

AV  $\Delta M$  6.72 PHASE SHIFTAV  $\Delta R$  1.87 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





@ 007		@ 117		@ 227		@ 297	
M1	R1	M2	R2	M3	R3	M4	R4
52.3	45.3	36.4	46.1	52.3	45.4	35.8	46.0
52.1	45.3	36.3	46.1	52.1	45.2	36.1	46.0
Av. 52.20	45.30	36.35	46.10	52.20	45.30	35.95	46.00

M1	52.20	R1	45.30
M2	36.35	R2	46.10
$\Delta M_{12}$	15.85	$\Delta R_{12}$	-0.80

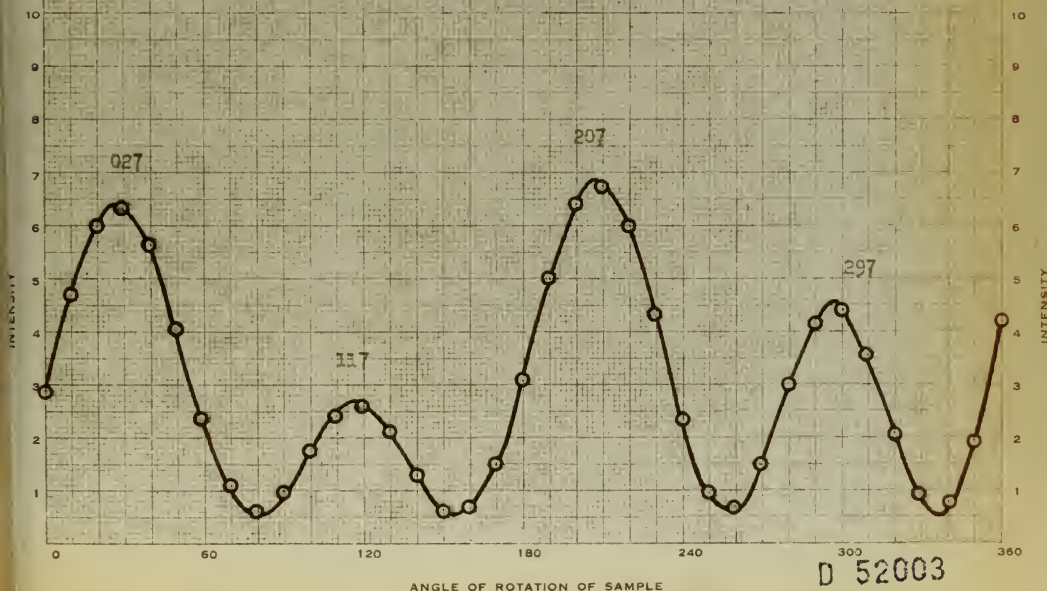
M3	52.20	R3	45.30
M4	35.95	R4	46.00
$\Delta M_{34}$	16.25	$\Delta R_{34}$	-0.70

Av  $\Delta M$  16.05 PHASE SHIFT

Av  $\Delta R$  -0.75 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





SAMPLE No 23

150 PPM  $\lambda_2$ 

DATE 15 JUL 68

CRYSTAL No. 3

SURFACE FILM CONDITION

2nd ELECTROPOLISH

@ 294		@ 024		@ 114		@ 204	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	P <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
39.1	44.5	45.8	46.4	39.0	44.5	45.5	46.1
39.0	44.5	45.7	46.2	39.1	44.6	45.5	46.4
AV. 39.05	44.55	45.75	46.30	39.05	44.55	45.50	46.25

M <sub>1</sub> 39.05	R <sub>1</sub> 44.55
M <sub>2</sub> 45.75	R <sub>2</sub> 46.30
$\Delta M_{12} -6.70$	$\Delta R_{12} -1.85$

M <sub>3</sub> 39.05	R <sub>3</sub> 44.55
M <sub>4</sub> 45.50	R <sub>4</sub> 46.25
$\Delta M_{34} -6.45$	$\Delta R_{34} -1.70$

AV  $\Delta M -6.58$ 

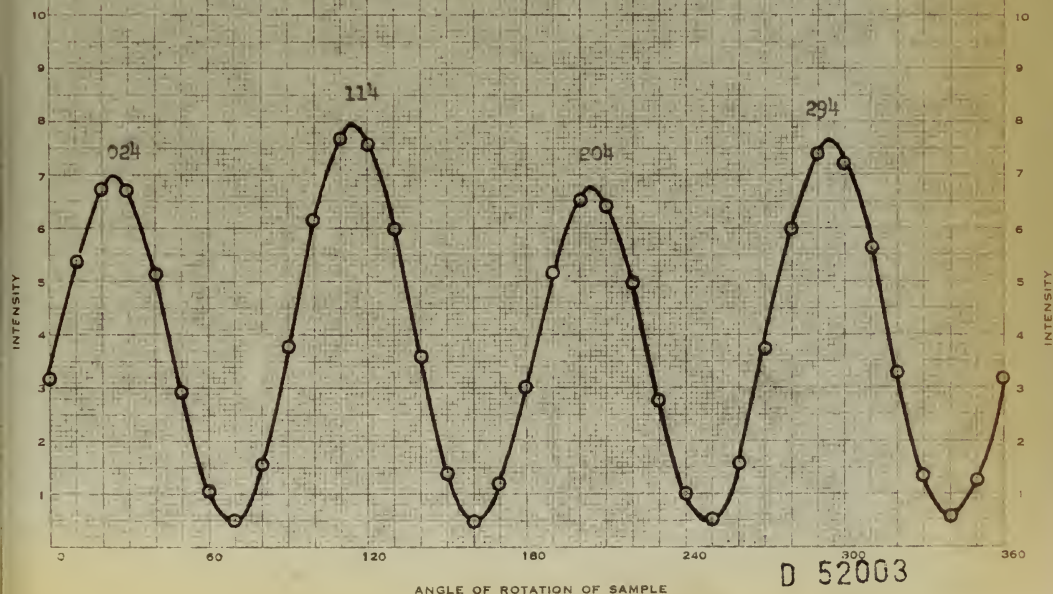
PHASE SHIFT

AV  $\Delta R -1.77$ 

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003

ANGLE OF ROTATION OF SAMPLE



CRYSTAL NO 3

SURFACE FILM CONDITION

1st AMPLITUDE 03 17 17

@ 044		@ 133		@ 223		@ 313	
M1	R1	M2	R2	M3	R3	M4	R4
32.4	40.8	51.6	39.3	32.0	41.6	51.8	39.2
32.0	41.0	51.4	39.6	32.0	41.2	51.6	39.0
AV 32.20	40.90	51.50	39.45	32.00	41.40	51.70	39.10

M1	32.20	R1	40.90
M2	51.50	R2	39.45
$\Delta M$	-19.30	$\Delta R$	-1.05

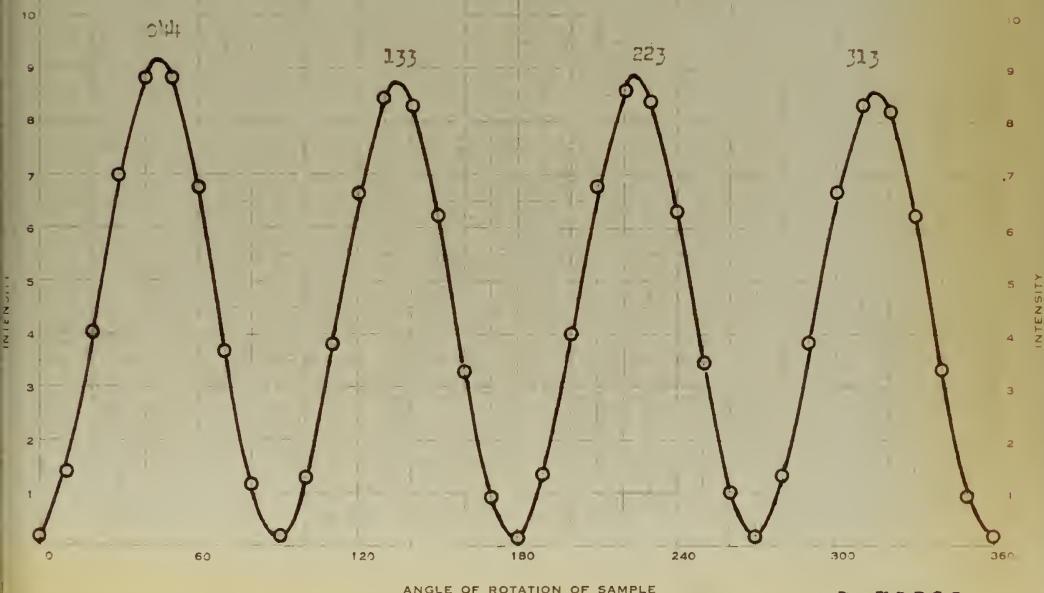
M3	32.00	R3	41.40
M4	51.70	R4	39.10
$\Delta M$	-19.70	$\Delta R$	-0.30

AV  $\Delta M$  -19.50 PHASE SHIFT

AV  $\Delta R$  1.37 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE No 2F

240 PPM  $N_2$ 

DATE 15 APRIL 1952 AM

CRYSTAL No 3

SURFACE FILM CONDITION

1st AUTOCLAVE 300°C at 15 MIN.

@ .375		@ .558		@ .168		@ .078	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
50.7	45.7	36.9	45.5	49.1	45.2	36.2	45.4
50.4	45.5	37.0	45.2	49.2	45.1	36.1	45.5
50.6	45.3						
AV. 50.57	45.50	36.95	45.35	49.15	45.15	36.15	45.45

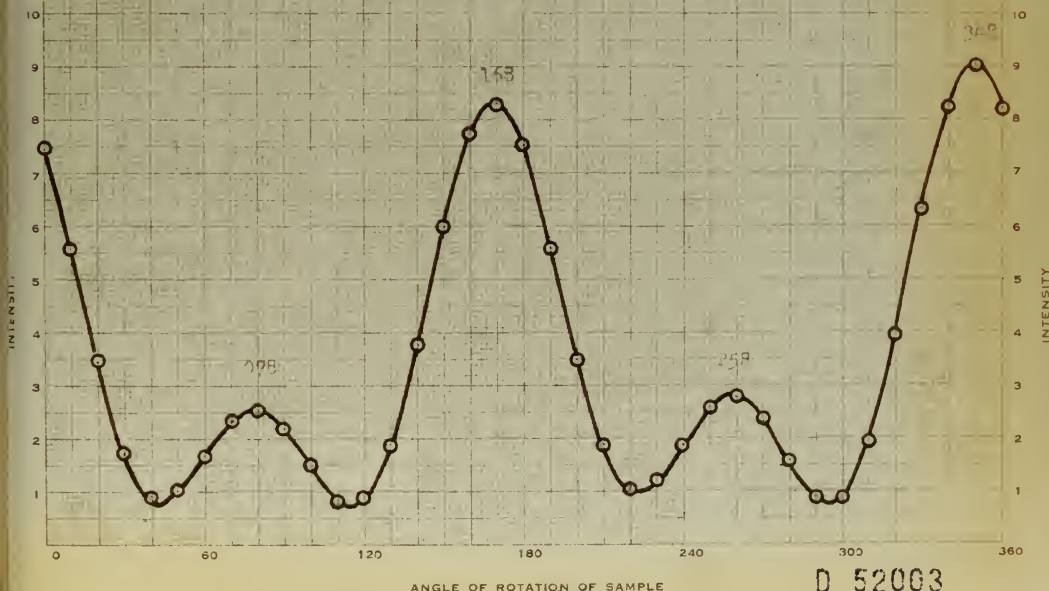
M <sub>1</sub>	50.57	R <sub>1</sub>	45.50
M <sub>2</sub>	36.95	R <sub>2</sub>	45.35
$\Delta M_{12}$	13.62	$\Delta R_{12}$	.15

M <sub>3</sub>	49.15	R <sub>3</sub>	45.15
M <sub>4</sub>	36.15	R <sub>4</sub>	45.45
$\Delta M_{34}$	13.00	$\Delta R_{34}$	-.30

AV  $\Delta M$  13.31 PHASE SHIFTAV  $\Delta R$  -0.08 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D 52003





CRYSTAL No. 3

SURFACE FILM CONDITION 2nd ELECTROPOLISH

@ 313		@ 323		@ 333		@ 343	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
46.1	46.5	39.7	44.7	46.5	46.6	39.5	44.8
46.6	46.6	39.6	44.7	46.3	46.6	39.3	44.8
Av. 46.55	46.55	39.65	44.70	46.40	46.60	39.40	44.80

M <sub>1</sub>	46.50	R <sub>1</sub>	46.55
M <sub>2</sub>	39.65	R <sub>2</sub>	44.70
$\Delta M_{12}$	6.85	$\Delta R_{12}$	1.85

M <sub>3</sub>	46.40	R <sub>3</sub>	46.60
M <sub>4</sub>	39.40	R <sub>4</sub>	44.80
$\Delta M_{34}$	7.00	$\Delta R_{34}$	1.80

Av  $\Delta M$  6.92

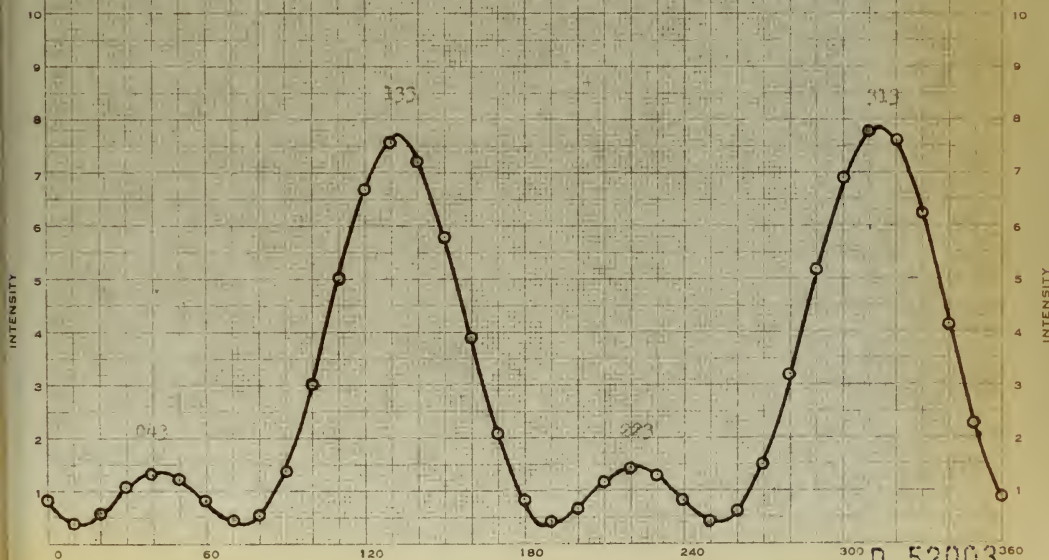
PHASE SHIFT

Av  $\Delta R$  1.82

PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE





SAMPLE NO 25

240 PPM  $N_2$ 

DATE 11 APRIL 1952 PM

CRYSTAL NO 4

SURFACE FILM CONDITION 2nd ELECTROPOLISH

@ 285		@ 196		@ 106		@ 616	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
43.5	45.8	41.3	45.0	43.6	45.8	41.2	45.1
43.5	45.9	41.3	45.0	43.5	45.9	41.1	45.1
Av. 43.55		41.30		43.55		41.15	
45.85		45.00		45.85		45.10	

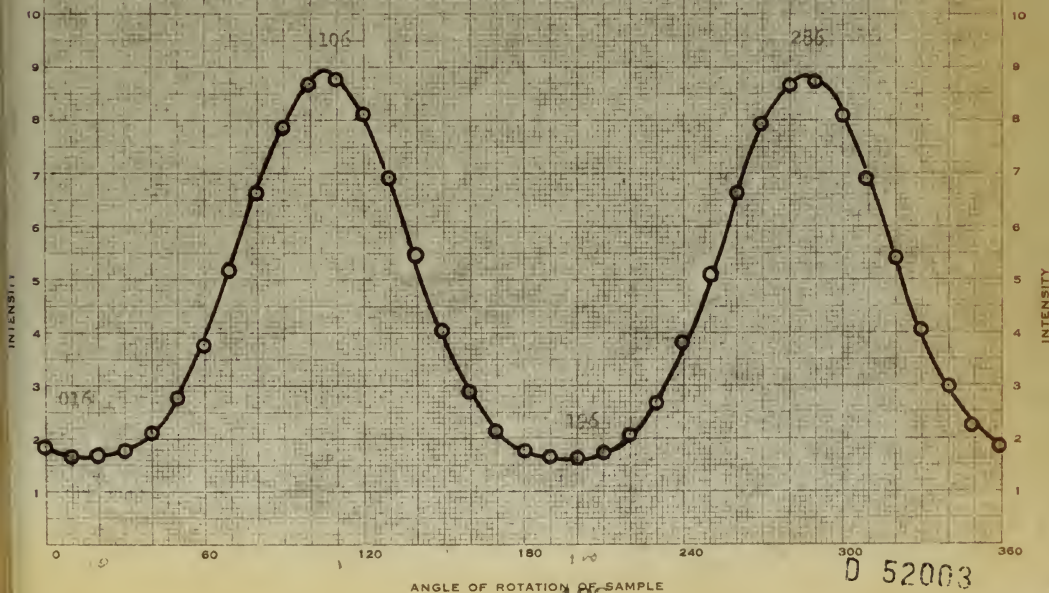
M <sub>1</sub>	43.55	R <sub>1</sub>	45.85
M <sub>2</sub>	41.30	R <sub>2</sub>	45.00
$\Delta M_{12}$	2.25	$\Delta R_{12}$	.85

M <sub>3</sub>	43.55	R <sub>3</sub>	45.85
M <sub>4</sub>	41.15	R <sub>4</sub>	45.10
$\Delta M_{34}$	2.40	$\Delta R_{34}$	.75

AV  $\Delta M$  2.32 PHASE SHIFTAV  $\Delta R$  0.80 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE







SAMPLE NO 2F

240 PPM N<sub>2</sub>

DATE 15 APRIL 1952 PM

CRYSTAL NO 4

SURFACE FILM CONDITION

1st AUTOCLAVE 15 MIN. @ 295°C

@ 324		@ 274		@ 144		@ 054	
M <sub>1</sub>	R <sub>1</sub>	M <sub>2</sub>	R <sub>2</sub>	M <sub>3</sub>	R <sub>3</sub>	M <sub>4</sub>	R <sub>4</sub>
43.7	48.2	41.6	43.3	41.7	48.5	41.8	42.9
43.9	48.2	41.5	43.4	41.5	48.5	41.6	43.1
AV. 43.80		41.55		41.60		41.70	
48.20		43.35		48.55		43.00	

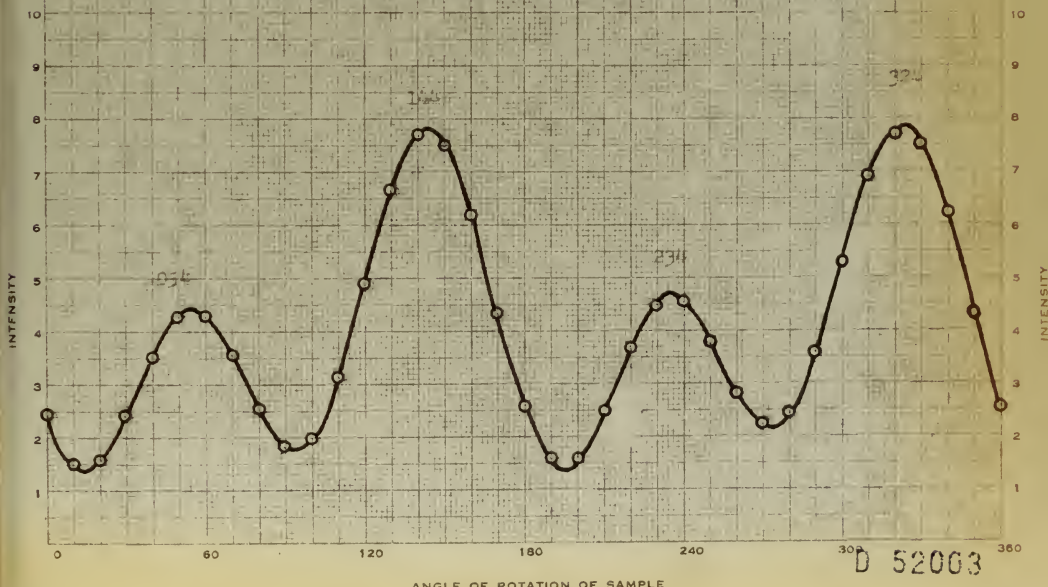
M <sub>1</sub>	43.80	R <sub>1</sub>	48.20
M <sub>2</sub>	41.55	R <sub>2</sub>	43.34
$\Delta M_{12}$	2.25	$\Delta R_{12}$	4.85

M <sub>3</sub>	41.60	R <sub>3</sub>	48.55
M <sub>4</sub>	41.70	R <sub>4</sub>	43.00
$\Delta M_{34}$	0.10	$\Delta R_{34}$	5.55

AV  $\Delta M$  2.58 PHASE SHIFTAV  $\Delta R$  5.20 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



ANGLE OF ROTATION OF SAMPLE

D 52003





CRYSTAL NO 4

SURFACE FILM CONDITION 3rd BLEND-POLISH

	@ 314		@ 224		@ 124		@ 044	
	M1	R1	M2	R2	M3	R3	M4	R4
	41.8	45.3	43.6	46.1	41.4	45.3	43.5	46.0
	41.6	45.3	43.6	46.0	41.6	45.3	43.4	46.0
AV.	41.70	45.30	43.60	46.05	41.50	45.30	43.45	46.00

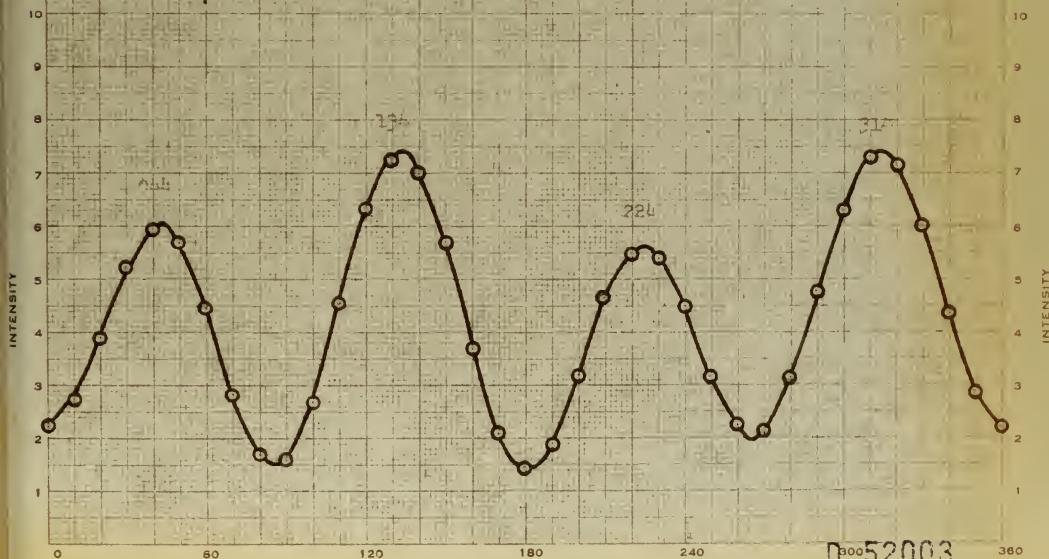
M1	41.70	R1	45.30
M2	43.60	R2	46.05
$\Delta M_{12}$	-1.90	$\Delta R_{12}$	- .75

M3	41.50	R3	45.30
M4	43.45	R4	46.00
$\Delta M_{34}$	-1.95	$\Delta R_{34}$	- .70

AV  $\Delta M$  -1.92 PHASE SHIFTAV  $\Delta R$  -0.72 PHASE ROTATION

PLOT THESE VALUES VERSUS CORROSION TIME

GRAPH OF POLARIZED LIGHT INTENSITY VERSUS ANGLE OF ROTATION OF SAMPLE



D30052003



Appendix E

Summary of Data

The data are summarized in four sets of curves as follows:

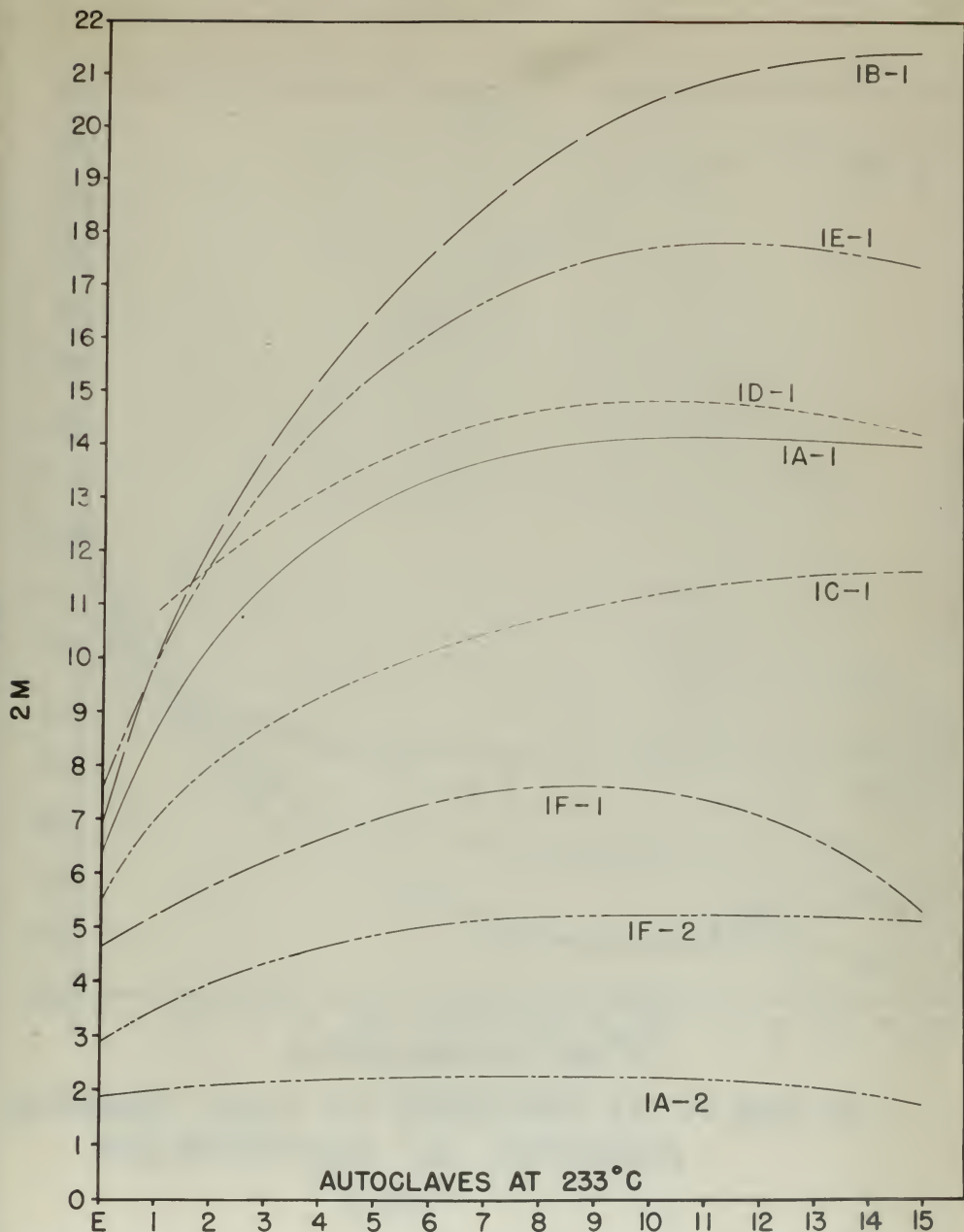
1. Run #1

- (a) Paired plot of phase shift versus number of autoclavings.
- (b) Paired plot of rotation of plane of polarisation versus number of autoclavings.

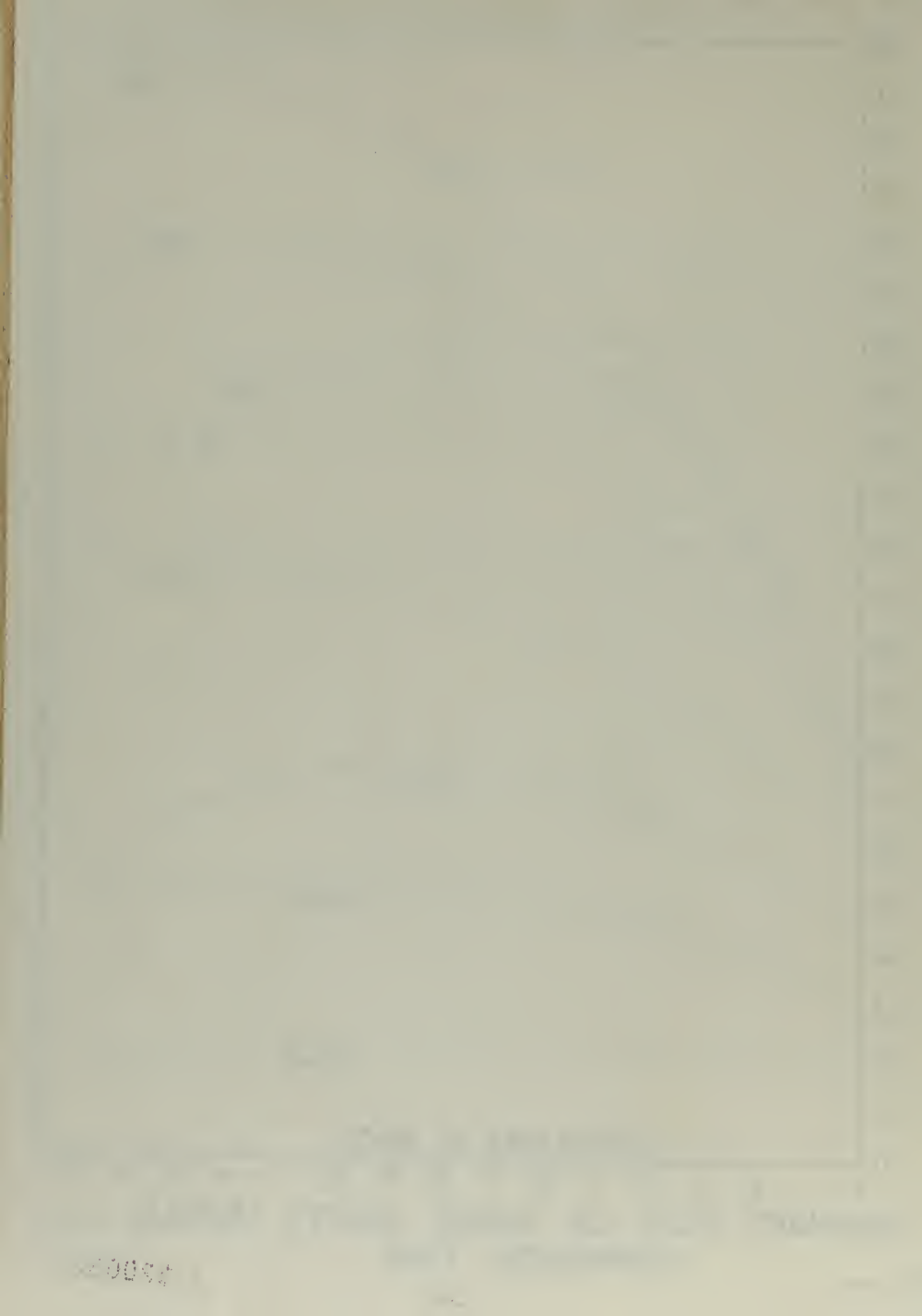
2. Run #2

- (a) Paired plot of phase shift versus number of autoclavings.
- (b) Paired plot of rotation of plane of polarization versus number of autoclavings.

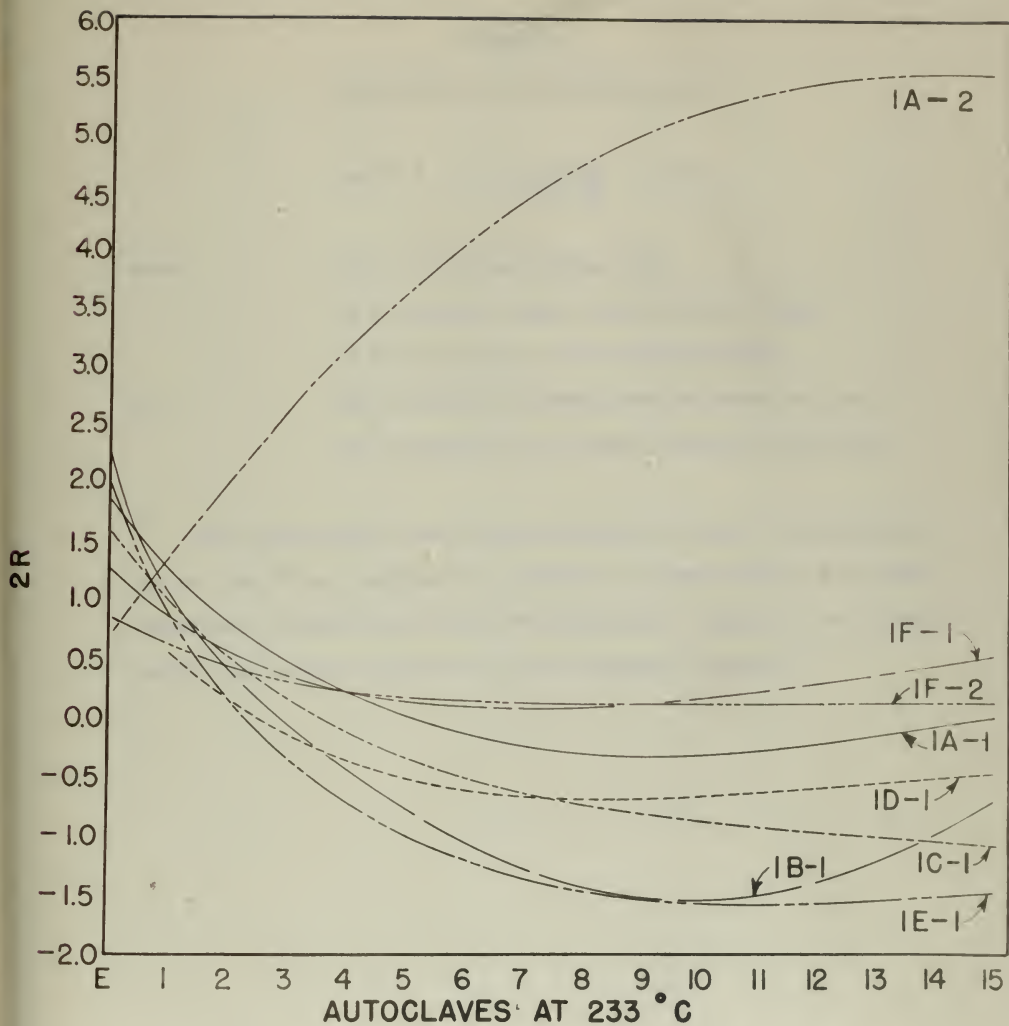




SUMMARY PLOT OF PHASE SHIFTS VERSUS CORROSION TIME







SUMMARY PLOT OF ROTATION OF PLANE OF  
POLARIZATION VS. CORROSION  
TIME



Appendix FCalculation of Characteristic Angle

$$\tan \tau = -0.6666 \frac{\sin 2m}{\sin 2r} + 0.0208$$

where

$\tau$  is the characteristic angle

$2m$  is the phase shift in the reflected light

$2r$  is the rotation in the reflected light

and

the constants are those given by Bausch and Lomb  
for Compensator No. 37 with Zirconarc white light.

Five place values for  $\sin 2m$  and  $\sin 2r$  were used in calculations.  $2m$ ,  $2r$ , and  $\tau$  are tabulated in Appendix F for each crystal in the electropolished condition and after each autoclaving. Plots of the characteristic angle versus corrosion time are contained in Chapter IV.

D 52003



RUN #1

	<u>141</u>			<u>142</u>		
	<u>2m</u>	<u>2r</u>	<u>L</u>	<u>2m</u>	<u>2r</u>	<u>L</u>
E	6.50	1.87	294-14	1.88	0.70	299-28
1	8.70	1.25	282-15	2.02	1.36	316-00
2	10.30	0.80	276-42	2.12	2.00	325-32
3	11.43	0.48	273-38	2.13	2.61	331-45
4	12.27	0.22	271-33	2.23	3.15	335-43
5	12.90	0.02	270-08	2.27	3.62	338-20
6	13.57	-0.12	269-13	2.30	4.05	337-43
7	13.70	-0.23	268-33	2.31	4.45	341-56
8	13.91	-0.30	268-08	2.32	4.77	343-05
9	14.03	-0.32	268-01	2.32	5.02	343-56
10	14.09	-0.30	268-09	2.30	5.21	342-31
11	14.10	-0.27	266-20	2.26	5.35	345-22
12	14.09	-0.21	268-42	2.18	5.45	346-11
13	14.05	-0.15	268-04	2.08	5.51	347-00
14						
15	13.95	0.00	270-00	1.80	5.55	348-46

D 52003

D 5200





RUN #1

	<u>1B1</u>			<u>1C1</u>		
	<u>2m</u>	<u>2r</u>	<u>T</u>	<u>2m</u>	<u>2r</u>	<u>T</u>
5	6.77	2.03	234-27	5.48	1.80	233-52
1	10.00	1.05	279-01	7.06	0.97	281-43
2	12.16	0.42	272-59	8.08	0.53	275-41
3	13.85	-0.03	268-08	8.85	0.17	271-40
4				9.35	-0.12	268-53
5	16.47	-0.77	265-56	9.75	-0.53	267-05
6	17.58	-1.07	264-43	10.14	-0.51	265-40
7	18.52	-1.28	264-00	10.47	-0.64	264-45
8	19.28	-1.42	263-36	10.76	-0.74	264-05
9	19.93	-1.53	263-15	11.00	-0.82	263-36
10	20.45	-1.55	263-24	11.18	-0.88	263-15
11	20.62	-1.51	263-37	11.33	-0.93	262-57
12	21.10	-1.40	264-12	11.45	-0.98	262-39
13	21.27	-1.23	264-57	11.55	-1.01	262-30
14						
15	21.45	-0.72	267-03	11.65	-1.08	262-05



RUN #1

	1D1			1d1		
	<u>2m</u>	<u>2r</u>	<u>12</u>	<u>2m</u>	<u>2r</u>	<u>12</u>
E	9.90	1.10	291-24	7.55	2.00	291 <sup>0</sup> -49'
1	10.87	0.55	274-22	9.95	.92	277 <sup>0</sup> -58'
2	11.73	0.15	271-06	11.90	.20	271 <sup>0</sup> -28'
3	12.50	-0.15	268-58	13.25	-.33	268 <sup>0</sup> -28'
4	13.13	-0.36	267-58	14.43	-.70	265 <sup>0</sup> -48'
5	13.67	-0.51	266-46	15.35	-.98	264 <sup>0</sup> -29'
6	14.10	-0.61	266-15	16.10	-1.18	263 <sup>0</sup> -40'
7	14.40	-0.67	265-58	16.67	-1.34	263 <sup>0</sup> -03'
8	14.62	-0.70	265-52	17.15	-1.44	262 <sup>0</sup> -44'
9	14.75	-0.70	265-62	17.50	-1.52	262 <sup>0</sup> -29'
10	14.79	-0.68	266-01	17.70	-1.57	262 <sup>0</sup> -20'
11	14.77	-0.65	266-11	17.80	-1.58	262 <sup>0</sup> -20'
12	14.70	-0.61	266-24	17.77	-1.57	262 <sup>0</sup> -21'
13	14.58	-0.57	266-37	17.68	-1.56	262 <sup>0</sup> -22'
14						
15	14.20	-0.48	267-04	17.35	-1.50	262 <sup>0</sup> -51'

D 52003



RUN #1

	1F1			1F2		
	<u>2m</u>	<u>2r</u>	<u>C</u>	<u>2m</u>	<u>2r</u>	<u>C</u>
E	4.65	1.25	292°-08'	2.90	.88	293°-26'
1	5.24	.85	283°-45'			
2	5.77	.55	278°-10'	3.95	.43	279°-18'
3	6.24	.55	274°-49'	4.32	.30	275°-58'
4	6.65	.21	272°-43'	4.62	.21	273°-55'
5	7.00	.12	271°-29'	4.85	.17	273°-01'
6	7.27	.09	271°-04'	5.01	.13	272°-14'
7	7.48	.09	271°-02'	5.12	.12	272°-01'
8	7.62	.10	271°-08'	5.18	.12	272°-00'
9	7.65	.12	271°-21'	5.25	.11	271°-40'
10	7.60	.17	271°-56'	5.24	.11	271°-40'
11	7.55	.22	272°-35'	5.23	.12	271°-59'
12	7.05	.30	273°-40'	5.21	.12	271°-59'
13	6.67	.37	274°-46'	5.18	.12	272°-00'
14						
15	5.30	.51	276°-15'	5.13	.13	272°-11'

D 52003





RUN #2

<u>2A1</u>						
	<u>2m</u>	<u>2r</u>	<u>C</u>	<u>2A2</u>		
				<u>2m</u>	<u>2r</u>	<u>C</u>
E	6.53	1.83	295°-00'	-1.65	0.68	238°-36'
1	17.88	-1.00	265°-08'	2.33	9.35	351°-41'
<u>2B3</u>						
				<u>2C3</u>		
E	6.45	1.65	291°-10'	4.70	1.62	297°-37'
1	14.92	-1.22	262°-57'	-16.33	2.35	257°-50'
<u>2D1</u>						
				<u>2E3</u>		
E	6.72	1.87	291°-24'	-6.58	-1.77	292°-10'
1	16.05	-0.75	265°-58'	-19.50	1.87	261°-41'
<u>2F3</u>						
				<u>2F4</u>		
E	6.92	1.82	291°-43'	2.32	0.80	297°-36'
1	13.31	-0.08	269°-29'	2.58	5.20	342°-45'

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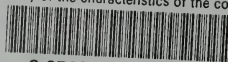
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